

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
27 September 2001 (27.09.2001)

PCT

(10) International Publication Number  
**WO 01/70842 A2**

- (51) International Patent Classification<sup>7</sup>: C08G 18/36, 18/32, C08J 9/00
- (21) International Application Number: PCT/US01/08888
- (22) International Filing Date: 20 March 2001 (20.03.2001)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:  
60/190,642 20 March 2000 (20.03.2000) US
- (71) Applicant: KT HOLDINGS, LLC [US/US]; 2137E Flinstone Drive, Tucker, GA 30084 (US).
- (72) Inventors: KATOOT, Mohammad, W. (deceased). KATOOT, Ahmed, M.; 1011 Lulworth Lane, Lawrenceville, GA 30044 (US).
- (74) Agents: ARNOLD, Jeffrey, B. et al.; Kilpatrick Stockton LLP, 2400 Monarch Tower, 3424 Peachtree Road, Atlanta, GA 30326 (US).
- (81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.
- (84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).
- Published:  
— without international search report and to be republished upon receipt of that report
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: POLYURETHANE FOAMS

(57) Abstract: Polyurethane foams are formed as the reaction product of a polyol selected from a fatty acid, a glycol, a mineral oil, a carbohydrate, or a combination thereof with a polyisocyanate in the presence of a catalyst and at least one blowing agent. Also, polyurethane foams are formed as the reaction product of a polyol selected from a vegetable oil, a mineral oil, a glycol, a syrup, or a combination thereof with a polyisocyanate in the presence of a catalyst and at least one blowing agent. A method for producing polyurethane foam compositions is directed to producing a polyol blend comprising a fatty acid, a glycol, a mineral oil, a carbohydrate, or a combination thereof, and reacting the polyol blend with a polyisocyanate blend in the presence of a catalyst and a blowing agent. Also, a method for producing polyurethane foam compositions is directed to producing a polyol blend comprising a vegetable oil, a mineral oil, a glycol, a syrup, or a combination thereof, and reacting the polyol blend with a polyisocyanate blend in the presence of a catalyst and a blowing agent.

WO 01/70842 A2

5

10

## POLYURETHANE FOAMS

## CROSS REFERENCE TO RELATED APPLICATION

The present invention claims priority from pending U.S. Provisional Patent Application Serial No. 60/190,642 entitled "Polyurethane Foams" filed March 20, 2000.

15

## FIELD OF THE INVENTION

The present invention relates generally to polyurethanes, and more specifically, the present invention relates to polyurethane foams and a process for preparing polyurethane foams.

20

## BACKGROUND OF THE INVENTION

Due to the finite supply of fossil fuels and the high cost of energy, the need to design energy-efficient buildings that are also economical becomes important. There are many conventional foams that are on the market, which are employed to improve the energy efficiency of buildings and structures. However, these foams, including polyurethane foams, use expensive chemicals in the manufacture of the foams, and many of these foams have toxic by-products after reaction.

25

30

Polyurethanes are formed by the reaction of a polyisocyanate compound, such as toluene diisocyanate (TDI) or diphenylmethane diisocyanate (MDI) with a polyhydroxyl-containing compound, such as a high molecular weight polyol. Generally, streams of approximately equal volume of the polyol and polyisocyanate are intermixed in a mixing

head and then dispensed into a cavity or mold or onto a surface. After the polyisocyanate and the polyol are mixed, typically in the presence of a catalyst, the hydroxyls of the polyol and isocyanates of the polyisocyanate react to form the polyurethane. Commonly employed catalysts include tertiary amines (e.g., diaminobicyclooctane and N,N,-  
5 dimethylaminoethanol), lead compounds (e.g., lead octoate), tin compounds (e.g., dibutyltin dilaurate and stannous carboxylates, such as stannous octoate). Absent a blowing agent, the polymerization reaction produces a solid polyurethane.

Polyurethane foams are made by forming gas bubbles in the polymerizing mixture to fill or expand the polyurethane foam cells, which is achieved by using a blowing  
10 agent. The density of such foams can be controlled by the quantity and efficiency of the blowing agents. As indicated in U.S. Patent No. 5,177,119 to *Motte*, water may be employed as a blowing agent. Water added to the polyol reacts with the isocyanates to provide CO<sub>2</sub> *in situ*, thereby causing the polyurethane to foam. Additionally, the use of low-boiling inert liquids, such as chlorofluorocarbons (CFCs), hydrochlorofluorocarbons (HCFCs),  
15 hydrofluorocarbons (HFCs), and pentanes to replace or augment the chemical blowing action of water has lead to advantages in certain properties, such as low thermal conductivity. Due to environmental concerns, however, CFCs, HCFCs, and HFCs are disfavored over foams blown only with CO<sub>2</sub>.

*Motte* states that the use of CO<sub>2</sub> generated from the reaction of water with  
20 isocyanate as a sole blowing agent requires relatively large amounts of water to obtain low density polyurethane foam grades. Such foams are difficult to process due to the increased exothermic conditions. Further, water also reacts with the polyisocyanate to produce a high density aromatic hard segment, such as a polyurea portion. With water levels above 4 parts by weight, this hard segment increase becomes a significant percentage that overpowers the  
25 softening characteristics of the standard polyols. To overcome this problem, it is typical to employ polyols or polyol blends having reduced functionality with relatively long polymer chain lengths. Here, functionality refers to the number of hydroxyl groups per molecule of the polyol available to react with the isocyanate groups.

The choice of polyol, especially the polymer length, size, flexibility of  
30 molecular structure, and functionality influences the degree of cross-linking achieved in the polyurethane. Further, the degree of cross-linking has an effect on the stiffness of the

polyurethane. For example, to obtain a rigid foam, there should be a stiff polymer network, and, hence a high degree of cross-linking. In contrast, for flexible foams there should be a proportionately lesser degree of cross-linking.

Since the functionality of either the long-chained, high molecular weight polyol or the isocyanate can be adjusted, a wide variety of branched or cross-linked polymers can be formed. The hydroxyl-containing component covers a wide range of molecular weights and types, including polyester and polyether polyols. The polyfunctional isocyanates can be aromatic, aliphatic, cycloaliphatic, or polycyclic in structure and can be used directly as produced or modified. This flexibility in the selection of reactants, although such reactants are relatively expensive, leads to a wide range of physical properties that allows polyurethane foams to play an important role in the world market for quality products from synthetic polymers.

The versatility of polyurethane foams is demonstrated by the following summary of some of the vast polyurethane foam applications:

- Automotive – seating, sound insulation, dashboards, steering wheels, interior trim;
- Appliances – insulation in refrigerators, freezers, chiller cabinets, and refrigerated vehicles;
- Composite wood products – construction panels and boards, packaging, playgrounds, and sports surfaces;
- Coatings and adhesives – used to protect a wide range of surfaces and objects in infrastructure applications, in the home, at work and leisure;
- Polyurethane adhesives – used to laminate flexible materials (textiles, paper, board etc.) for bonding rubbers, textiles, leather, and the like and for bonding metal to metal, rubber, and plastics;
- Construction – insulation panels for walls (internal and external) and roofs, both domestic and commercial, cavity insulation, spray applied roof insulation, and pre-fabricated insulation for tanks and pipes;
- Footwear – sole cushions; and
- Seat cushions – cushions and backs for domestic and office chairs and sofas, mattresses, pillows, and imitation wood.

Despite existing polyurethane foams and methods of making such polyurethane foams, there is a need for polyurethane foams and a method of making polyurethane foams without the need to employ hydrocarbon-based polyols. Further, there is a need for polyurethane foams and a method of making polyurethane foams from relatively inexpensive components. It is to the provision of such polyurethane foams and method of making that meets these needs that the present invention is primarily directed.

## 10 SUMMARY OF THE INVENTION

The polyurethane foams of the present invention can be used in many applications. One of the principal uses of the polyurethane foams of the present invention is insulation. The present environment is concerned with global warming, heat conservation, and reduced CFC's and HCFC's. The present invention is specifically designed and formulated to replace all currently available expensive hydrocarbon based polyols with relatively low cost, naturally occurring and readily available oils and carbohydrates. The present invention is designed as an essential part of any construction that values long-term energy savings and acoustic shielding. The foam produced according to the present invention flows easily to fill the area regardless of shape or the presence of obstructions such as pipes, wires, and electrical boxes.

In one aspect of the present invention, the sprayed polyurethane foam of the present invention is applied to roofing as a liquid, expanding approximately some 40 times its original liquid volume, and can be used to fill voids, cracks, and crevices as well as providing an air-tight, weatherproof membrane for the roof. The foam dries in seconds following application and fully adheres to the substrate. Due to the lightweight of the foam, it adds very little additional weight to the roof. The versatility of the polyurethane foam lends itself to on-site applications. Residential, commercial, and industrial constructions are all candidates for polyurethane foam applications. In addition, the foam adds strength to metal and wood stud cavities due to excellent adhesion and strength to weight ratios.

Sprayed polyurethane foam (SPF) can provide better climate control within a building envelope. Better climate control reduces the consumption of fossil fuels, thereby

reducing greenhouse gases released into the atmosphere. Further, SPF climate control ability enables the downsizing of the heating and cooling equipment of a building, further reducing energy usage.

SPF provides a continuous air barrier preventing moisture infiltration through air leakage, minimizing dew point problems and condensation within the building envelope, avoiding thermal bridging, resisting heat movement in all directions, and providing reliable performance under varying conditions. By controlling moisture infiltration, SPF also provides greater durability to buildings. The number one cause of building deterioration is moisture within a building envelope.

The present invention also has applications as a two-component polyurethane construction low rise expanding adhesive, which can be utilized for bonding membranes or insulation type materials to a variety of substrates. Compatible deck substrates include concrete, gypsum, cellular LWC.

Accordingly, it is an object of the present invention to provide a method and composition for producing foam having high insulation values and being produced from relatively low cost, naturally occurring and readily available oils and carbohydrates.

It is a further object to provide a foam, which is made from materials, which are not irritating to occupants of structures in which the foam is used.

It is a still further object of this invention to provide a foam and a process for making a foam, which can be easily used to install the foam in a structure.

It is a yet another object of this invention to provide a foam and a process for making a foam which is flame retarding.

These and other object, features and advantages of the present invention will become apparent after a review of the following detailed description of the disclosed embodiments.

The foam may also be tailored to variable densities, cell structures, tensile strengths and other desired physical properties. The polyurethane resins can be produced in varying forms due to properties that exhibit high elastic modulus, good electrical resistance, and high moisture-proof crystalline structures.

## DETAILED DESCRIPTION OF THE INVENTION

According to the present invention, polyurethanes are produced using a two-part reactive system. The first reactive part may contain one or more organic isocyanates, as well as, other components such as initiators and/or catalysts commonly referred to as the "A" side. In one aspect of the invention, the organic isocyanate comprises polymeric 4,4'-diphenyl methane diisocyanate. The isocyanate concentration is calculated at 90-120% of the theoretical amount required to react with both hydroxyl groups and water. This percentage is known as the isocyanate index. The second reactive part, commonly referred to as the "B" side, may contain one or more compounds which contain functional groups that react with organic isocyanates to form a polyurethane. The urethane foams are formed by the process of simultaneous polymerization and expansion. The gas for expansion is primarily carbon dioxide, CO<sub>2</sub>, formed by the reaction of isocyanate and water. Carbon dioxide can also be used as an effective blowing agent, as well as high-pressure air and conventional low boiling compounds, to complete the formation of the foam cell structure and to prevent shrinkage of the cell.

Polyurethane foams in accordance with the present invention comprise the reaction product of a polyol selected from a fatty acid, a glycol, a mineral oil, a carbohydrate, or a combination thereof with a polyisocyanate in the presence of a catalyst and at least one blowing agent. Fatty acids include, but are not limited to, palmitic, stearic, oleic linoleic, myristic, arachidic, ricinoleic, or a combination thereof. The term glycol comprises glycol, ethylene glycol, a polyethylene glycol, diethylene glycol, dipropylene glycol, propylene glycol, hexylene glycol, neopentyl glycol, bisphenol A, 2-methyl propanediol, trimethylolpropane, 1, 4-butane diol, or a combination thereof. The polyethylene glycol has a molecular weight range between about 200 and about 600. Carbohydrates include polyhydric alcohols having the general formula CH<sub>2</sub>OH(CHOH)<sub>n</sub>CH<sub>2</sub>OH, wherein n is selected from 2 to 5. Examples of polyhydric alcohols include, but are not limited to sucrose, glucose, fructose kojibiose, turanose, isomaltose, maltose, dextrose, glycerol, and combinations thereof. Blowing agents can be selected from water, a low-boiling inert liquid, carbon dioxide, air, gasses which are inert with respect to the polyurethane or components of the polyurethane, or a combination thereof. Examples of the low-boiling inert liquid include, but are not limited to chlorofluorocarbon, a hydrochlorofluorocarbon, a hydrofluorocarbon, a

pentane, chloroform, or a combination thereof. On the A side, polyisocyanates include hexamethylene diisocyanate, toluene 2-4 diisocyanate, naphthalene 1,5 diisocyanate, diphenylmethane 2,4' diisocyanate, 4,4' diphenyl methane diisocyanate, or a combination thereof. However, other polyisocyanates employed in the art may likewise be employed with  
5 the present invention.

Yet, in another aspect of the present invention, polyurethane foams in accordance with the present invention comprise the reaction product of a polyol selected from a vegetable oil, a mineral oil, a glycol, a syrup, or a combination thereof with a polyisocyanate in the presence of a catalyst and at least one blowing agent. Vegetable oil  
10 includes, but not limited to, canola oil, sesame oil, sunflower oil, soybean oil, palm oil, castor oil, coconut oil, cotton seed oil, olive oil, apricot oil, avocado oil, safflower oil, linseed oil, coconut oil, corn oil, babassu oil, tung oil, perilla oil, oiticica oil, a hydrogenated derivative thereof, or a combination thereof. Syrup includes any syrup comprising a sugar, such as, sucrose, glucose, fructose kojibiose, turanose, isomaltose, maltose, dextrose,  
15 glycerol, and combinations thereof. Such syrups include, but are not limited to maple syrup, honey, corn syrup, cane syrup, golden syrup, molasses, sorghum, beet syrup, or a combination thereof.

Still, another aspect of the present invention is directed to a method for producing polyurethane foam compositions. This method comprises producing a polyol  
20 blend comprising a fatty acid, a glycol, a mineral oil, a carbohydrate, or a combination thereof, and reacting the polyol blend with a polyisocyanate blend in the presence of a catalyst and a blowing agent. The polyol blend and the polyisocyanate blend can be reacted with one another in equal parts by weight, or in the ratios described below.

Another aspect of the present invention is directed to a method for producing  
25 polyurethane foam compositions in accordance with the present invention. The method comprises producing a polyol blend comprising a vegetable oil, a mineral oil, a glycol, a syrup, or a combination thereof, and reacting the polyol blend with a polyisocyanate blend in the presence of a catalyst and a blowing agent.

Fatty acids include, but are not limited to, palmitic, stearic, oleic linoleic,  
30 myristic, arachidic, ricinoleic, or a combination thereof. Vegetable oil includes, but not limited to, canola oil, sesame oil, sunflower oil, soybean oil, palm oil, castor oil, coconut oil,



cotton seed oil, olive oil, apricot oil, avocado oil, safflower oil, linseed oil, coconut oil, corn oil, babassu oil, tung oil, perilla oil, oiticica oil, a hydrogenated derivative thereof, or a combination thereof. Syrup includes any syrup comprising a sugar, such as, sucrose, glucose, fructose kojibiose, turanose, isomaltose, maltose, dextrose, glycerol, and combinations thereof. Such syrups include, but are not limited to maple syrup, honey, corn syrup, cane syrup, golden syrup, molasses, sorghum, beet syrup, or a combination thereof. Carbohydrates include monosaccharides, disaccharides, glycerides, polyhydric alcohols, and combinations thereof. Polyhydric alcohols have the general formula  $\text{CH}_2\text{OH}(\text{CHOH})_n\text{CH}_2\text{OH}$ , wherein  $n$  is selected from 2 to 5. Examples of polyhydric alcohols include, but are not limited to sucrose, glucose, fructose kojibiose, turanose, isomaltose, maltose, dextrose, glycerol, glyceride and combinations thereof.

The term glycol comprises glycol, ethylene glycol, a polyethylene glycol, diethylene glycol, dipropylene glycol, propylene glycol, hexylene glycol, neopentyl glycol, bisphenol A, 2-methyl propanediol, trimethylolpropane, 1, 4-butane diol, or a combination thereof. The polyethylene glycol has a molecular weight range between about 200 and about 600.

Blowing agents can be selected from water, a low-boiling inert liquid, carbon dioxide, air, gasses which are inert with respect to the polyurethane or components of the polyurethane, or a combination thereof. Examples of the low-boiling inert liquid include, but are not limited to chlorofluorocarbon, a hydrochlorofluorocarbon, a hydrofluorocarbon, a pentane, chloroform, or a combination thereof. On the A side, polyisocyanates include hexamethylene diisocyanate, toluene 2-4 diisocyanate, naphthalene 1,5 diisocyanate, diphenylmethane 2,4' diisocyanate, 4,4' diphenyl methane diisocyanate, or a combination thereof. However, other polyisocyanates employed in the art may likewise be employed with the present invention.

Further, the polyurethane foams of the present invention may include one or more resins, fillers, surfactants, stabilizers, cross-linkers, drying agents, stabilizers, preservatives, fire retardants, antibacterial agents, and any combination thereof.

The initiators and/or catalysts used in the present invention may be employed to control the polymerization and blowing processes. This control is important for polyurethane reactant foam systems. Imbalance between polymerization and foam

expansion can lead to foam collapse, serious imperfections, and cells that prematurely open, causing shrinkage of the cured foam and loss of adhesion properties.

The amount of polymers, carbohydrates, initiators, and catalysts may vary. Desirably the polymer, its initiators, carbohydrates, and catalysts comprise from about 40 to about 70 weight percent on the B side and the functional group on the A side from about 60 to 30 weight percent based upon the total combined weight of the mixture. Even more desirably the polymer comprises from about 55 to about 65 weight percent on the B-side and the functional group on the A side from about 45 to about 35 weight percent based upon the total combined weight of the mixture.

The production of the A side and the B side components, once completed can be easily mixed, the resulting liquid mixture can be sprayed or molded into the desired shape or form. The flexible polyurethane reactant foam system can be modified to produce various densities, strengths and cell structures. The procedures that can be used are similar to those employed utilizing industrial hydrocarbon polyols.

The flexible urethane foam systems of the present invention may employ suitable methacrylate and acrylate monomers in the foam formulations. In one aspect of the present invention, the two-component urethane foam system comprises, on the A side, an admixture of 4,4' diphenyl methane diisocyanate (MDI) and dimethyl sulfoxide (DMSO). In this embodiment 4,4' diphenyl methane diisocyanate, (MDI) and dimethyl sulfoxide (DMSO) are present, but not limited to, a weight ratio of approximately 78:22. In the present invention the use of reactant fillers that make up the two-component foam system comprise, on the B side, carbohydrates comprising, but not limited to, glucose, dextrose, and lactose, and fatty acids comprising, but not limited to, castor oil and linseed oil. As indicated in Example 1 below, glucose and lactose are present, but not limited to, a weight ratio of 87:13. The monomers, catalysts, and initiators that makeup the two component foam system comprise, on the B side, aqueous ammonia, a carbonyl group (e.g., an organic acid such as acetic acid), and dibutyltin dilaureate. In this embodiment these monomers, catalysts, and initiators are present, but not limited to, a weight ratio of 31:61:8.

In other aspects of the invention, the A side and the B side of a liquid, two-component polyurethane system comprises a compound or mixture of compounds containing functional groups which react with isocyanates. Castor oil, a trishydroxy fatty triglyceride or

ricinoleic acid, is an example of a naturally occurring polyol. It produces a low cost urethane ingredient. In these embodiments castor oil is combined with vinyl ester resins and various filler materials, such as calcium oxides, calcium carbonates along with one or more initiators and/or catalysts. (See Examples 2-9.) In the present invention CaO also acts as a catalyst.

5 The amount of castor oil, vinyl resins, fillers, initiators, and catalysts may vary. The isocyanate initiator of the foam is the final ingredient of the mixture.

In another aspect of the present invention, the A side and the B side of a liquid, two-component polyurethane system comprises a compound or mixture of compounds containing functional groups which react with isocyanates. These are carbohydrate products of photosynthesis comprising one, two, or more saccharose groups. The monosaccharide  
10 sugars (often called simple sugars) are composed of 2-7 carbon atoms. One of the carbons carries aldehydic or ketonic oxygen. The remaining carbons usually have hydrogen atoms and hydroxyl groups. Chief among the monosaccharides are glucose and fructose. Among the disaccharides are sucrose (cane and beet sugar) and lactose. Each of these carbohydrates  
15 produces a low cost urethane ingredient. The isocyanate initiator of the foam is the final ingredient of the mixture.

Polyisocyanates which can be used in the present invention include MDI, hexamethylene diisocyanate (HDI), toluene 2-4 diisocyanate (2-2-TDI), naphthalene 1,5 diisocyanate (NDI), diphenylmethane 2,4' diisocyanate (2,4'MDI), and combinations  
20 thereof. The present invention can use other ranges of isocyanates as are commonly available from manufacturers such as, BASF, Dow Chemical Company, Mobay, and Rubicon, to name only a few.

A variety of polyurethane foams of differing rigidities and densities were prepared from different catalysts initiators, fillers, and varying amounts of these ingredients  
25 as exhibited in the examples below. The resulting ingredients were combined in plastic containers, and thoroughly mixed. The compositions were allowed to expand freely and left to cure.

Applications of the present invention are described below. However, these applications are only a few of the vast applications of the polyurethane foams of the present invention and are  
30 not to be deemed as limiting.

### Roofing

Sprayed polyurethane foam is applied as a liquid filling cracks and crevices, then expanding approximately 40 times its original liquid volume. The polyurethane foam dries within seconds after applied to the roof surface. Its expansion results in a weather tight roofing membrane that is fully adhered to the substrate. Because of polyurethane foam's lightweight, it adds little additional weight to the structure and is often used in remedial applications. Polyurethane foam has a history of more than 30 years as a maintainable roofing medium. Polyurethane foam adds excellent insulation value to the structure and utility bills can reflect the difference.

### Construction

The versatility of polyurethane foam lends itself to on-site applications. This is of particular benefit since it can be considered during many phases of construction. Agricultural, poultry, meat packaging plants, cold storage warehousing facilities, and masonry cavities are typical examples of spray and pour polyurethane foam applications. Polyurethane foam adds strength to wood or metal stud cavities of commercial and residential buildings due to excellent adhesion and high strength to weight ratio. Polyurethane foams monolithic sealing capabilities can provide considerable utility savings. The list of such applications for polyurethane foam is extensive.

### Cavity Filling

Polyurethane foam is used widely in applications requiring injection and open cavity filling. The versatility of the product permits tailoring of the formulation to meet specific requirements. Depending on the needs, it can serve as an excellent insulation medium or enhance the structural integrity of the finished product. The list of applications is too many to mention and new opportunities develop constantly.

### Perimeter Wall Insulation

Polyurethane foam can be sprayed or poured to meet most application requirements and provide a monolithic weather tight seal. Sprayed or poured polyurethane foam or a combination of both are successfully used in the construction of cold storage

warehouses, atmosphere controlled buildings, and residential and commercial structures. With utility savings being a primary consideration, the added benefits of polyurethane foam's lightweight and excellent adhesion has introduced many new concepts in building design.

## 5 Corrosion Protection

A new generation of high performance plural component coatings are satisfying old problems while opening many new markets. These high performance coatings are polyureas, polyurethanes, and blended hybrids. Resistant of many solvents and chemicals, they dry rapidly and can be applied to various substrates. Underground pipe restoration as well as new pipe coating are further uses of these high performance coatings, demonstrating their excellent physical properties and diversity.

## Secondary Containment

High performance plural component coatings are a rapidly growing technology. These new coatings, polyureas, polyurethanes, and blended hybrids, have opened new markets and can be applied to a variety of substrates. Secondary containment is an example of a application for the polyurethane foams of the present invention, which has been a difficult problem to solve. Underground pipe, manhole cover restoration, parking decks, coating of new pipe, and waterproofing applications are further uses of these high performance materials. Producers report the rapid growth of these high performance coatings is attributed to formulation versatility and outstanding physical properties.

## Spray Molding

Polyurethanes and polyureas have very fast reaction times. These materials are dry to the touch within seconds after application reducing de-mold time dramatically. The producer to meet application requirements can alter physical properties of these materials.

## Insulation

One of the most efficient insulation materials for housing, commercial refrigeration, perimeter wall, masonry cavity fill and numerous other applications is

polyurethane foam. On-site applied as sprayed or poured liquid, polyurethane foam fills cracks and crevices, providing sealing capability. Polyurethane foam expands up to thirty times its liquid volume immediately upon application and is dry to the touch in seconds. The unique application process has an excellent history as a spray applied remedial roofing membrane sealant and insulation system. And because of polyurethane foams high insulating value, it out-performs many conventional insulations applied in equal thickness. Its high strength to low weight ratio has attracted architects and building consultants throughout the world.

#### 10 Entertainment Sets

Polyurethane foams' vast versatility lends themselves to on-site or studio production of needed settings. The savings can be considerable when compared to the cost involved to shoot the actual setting. Polyurethane foams of the present invention can be formulated to provide physical properties for structural applications, such as, facades to lightweight boulders to the replication of tree bark, to exacting duplications of extinct animals, to name only a few, are possible with polyurethane foams. Also, polyurethane foams have excellent weatherability and superior abrasion and impact resistance.

#### Marine Applications

Polyurethane foam is used in many marine applications. The high strength to weight ratio plus its resistance to hydrocarbons, such as oil and gasoline, make it an exceptional candidate for flotation applications. The adhesive qualities are particularly attractive. A polyurethane foam with a two pound density will support around 60 pounds of dead weight in water.

#### Adhesives

Polyurethane adhesives are employed to laminate flexible materials, such as, textiles, paper, board, etc., for bonding rubbers, textiles, leather, and the like and for bonding metal to metal, rubber, and plastics.

In accordance with the present invention, polyurethane foam compositions are disclosed for insulation foam, roofing foam, molding foam, hard foam, and adhesive foam.

However, any of these formulations are adaptable for other polyurethane foam uses not specified. Accordingly, these categories or designations should not be considered as limiting. In each of the following polyurethane foam compositions, the side A and side B components are admixed in separate mixing vessels. There is no requirement for the components to be added to the respective mixing vessels in the order listed. Rather, the side A and side B components can be added to the respective mixing vessels any order. However, the side A solutions and the side B solutions should not be introduced with one another until it is desired to produce the polyurethane foam. Further, the components can be thoroughly mixed as they are added or thoroughly mixed after all selected components are placed into the mixing vessel. After the side A and side B components, respectively, are admixed into solution, the side A and side B solutions are admixed and allowed to react with one another to form the polyurethane foam of the present invention. In one aspect of the present invention, the polyurethane foam comprises from about 65 to about 35 weight percent of the side A solution and from about 35 to about 65 weight percent of the side B solution. In another aspect of the present invention, the polyurethane foam comprises about 50 weight percent of the side A solution and about 50 weight percent of the side B solution. Mixing of the side A and side B solutions can occur within a vessel, a spray wand, a mold, a cavity, or any conventional mixing apparatus or device. Compositions of the polyurethane foams of the present invention are as follows.

### Insulation Foam

Insulations foams of the present invention have a density of about 0.5 pounds per cubic foot; however, the density of the insulation foam can be greater or less than 0.5 pounds per cubic foot. The following side A solution and side B solution, respectively, are admixed in separate mixing vessels, and thereafter mixed with one another in the amounts by weight as indicated above to form a polyurethane insulation foam.

#### Side A Components:

<u>Wt. %</u>	<u>Component</u>
50-100	4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)
0-50	1-vinyl-2-pyrrolidinone (NVP)
0-50	Dimethyl Sulfoxide (DMSO)
0-20	Silicon surfactant
0-50	FYROL™ (tri (2-chloroethyl)phosphate flame (retardant)
0-1	Antimicrobial/Antibacterial agent

## Side B Components:

	Wt. %	Component
	50-90	Carbohydrate
5	0-20	1-vinyl-2-pyrrolidinone (NVP)
	0-20	Dimethyl Sulfoxide (DMSO)
	0-50	Ethylene Glycol (EG)
	0-50	A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO <sub>2</sub> (20-21 wt. %) (EG-ST)
10	0-50	Polyethylene Glycol, 200 molecular weight (PEG200)
	0-50	Polyethylene glycol 400 molecular weight (PEG400)
	0-50	Polyethylene glycol 600 molecular weight (PEG600)
	0-30	FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
	0-30	Triethanolamine (TEOA)
15	0-10	Silicon surfactant
	0-2	Dibutyltin dilaurate (Di-10)
	0-10	2,2'-oxybis(N,N-dimethylethanamine) (CAS No. 3033-62-3) (ZF-20)
	0-10	Triethylamine (TEA)
	0-10	aminified sugar
20	0-6	N,N-dimethylaniline (N,N DMA)
	0-10	Carboxymethyl cellulose/polyacrylic acid aqueous solution
	0-10	Tripropylamine (TPA)
	0-10	Colloidal Silica (SNOWTEX)
	0-1	Antimicrobial/Antibacterial agent
25		

ROOFING FOAM

Roofing foams of the present invention are mostly closed cell (about 85% to about 95 %), are sprayable, and have a density from about 1 to about 4 pounds per cubic foot; however, the density of the roofing foam can be greater or less than this range. The following side A solution and side B solution, respectively, are admixed in separate mixing vessels, and thereafter mixed with one another in the amounts by weight as indicated above to form a polyurethane roofing foam.

## Side A Components:

	Wt. %	Component
35	50-100	4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)
	0-50	1-vinyl-2-pyrrolidinone (NVP)
	0-50	Dimethyl Sulfoxide (DMSO)
	0-20	Silicon surfactant
40	0-50	FYROL™ (tri(2-chloroethyl)phosphate flame retardant)



## Side B Components:

	<u>Wt. %</u>	<u>Component</u>
	40-90	Castor Oil
	0-40	Linseed Oil
5	0-50	Mineral Oil (heavy mineral oil CAS #8012-95-1, light mineral oil CAS #8042-47-5, or a combination thereof)
	0-50	Glycerol
	0-30	Polyethylene glycol/dodecylbenzenesulfonic acid solution
	0-50	Ethylene Glycol (EG)
10	0-50	A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO <sub>2</sub> (20-21 wt. %) (EG-ST)
	0-50	Polyethylene Glycol 200 molecular weight (PEG200)
	0-50	Polyethylene Glycol 400 molecular weight (PEG400)
	0-50	Polyethylene Glycol 600 molecular weight (PEG600)
15	0-30	FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
	0-30	Triethanolamine (TEOA)
	0-10	Silicon surfactant
	0-4	Dibutyltin dilaurate (Di-10)
	0-10	Dipropylene glycol (33LV Catalyst)
20	0-10	Bis(dimethyl amino propyl)methyl amine (POLYCAT 77)
	0-10	1,8-diazabicyclo(5,4,0)-7undecane 2-ethyl hexoate (POLYCAT 102)
	0-10	2,2'-oxybis(N,N-dimethylethanamine) (CAS No.- 3033-62-3) (ZF-20)
	0-10	Triethylamine (TEA)
	0-10	Aminified sugar
25	0-10	Carboxymethyl cellulose/polyacrylic acid aqueous solution
	0-10	Tripropylamine (TPA)
	0-10	Colloidal Silica (SNOWTEX)
	0-1	Antimicrobial/Antibacterial agent

30 Molding Foam

Molding foams of the present invention are mostly closed cell (about 85% to about 95 %) and have a density from about 1 to about 4 pounds per cubic foot; however, the density of the molding foam can be greater or less than this range. The following side A solution and side B solution, respectively, are admixed in separate mixing vessels, and thereafter mixed with one another in the amounts by weight as indicated above to form a polyurethane molding foam. The molding foam can be employed similarly as a resin to form molded objects.

## Side A Components:

	<u>Wt. %</u>	<u>Component</u>
40	50-100	4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)
	0-50	1-vinyl-2-pyrrolidinone (NVP)
	0-50	Dimethyl Sulfoxide (DMSO)
	0-20	Silicon surfactant

0-50 FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

Side B Components:

	Wt. %	Component
5	40-90	Castor Oil
	0-40	Linseed Oil
	0-50	Mineral Oil (heavy mineral oil CAS #8012-95-1, light mineral oil CAS #8042-47-5, or a combination thereof)
	0-50	Glycerol
10	0-30	Polyethylene glycol/dodecylbenzenesulfonic acid solution
	0-50	Ethylene Glycol (EG)
	0-50	A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO <sub>2</sub> (20-21 wt. %) (EG-ST)
	0-50	Polyethylene Glycol, 200 molecular weight (PEG200)
15	0-50	Polyethylene Glycol, 400 molecular weight (PEG400)
	0-50	Polyethylene Glycol, 600 molecular weight (PEG600)
	0-30	FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
	0-30	Triethanolamine (TEOA)
20	0-10	Silicon surfactant
	0-30	Calcium oxide (CaO)
	0-20	Aluminum trihydrate (ATH)
	0-50	CaCO <sub>3</sub>
25	0-4	Dibutyltin dilaurate (Di-10)
	0-10	Dipropylene glycol (33LV Catalyst)
	0-10	Bis(dimethyl amino propyl)methyl amine (POLYCAT 77)
	0-10	1,8-diazabicyclo(5,4,0)-7undecane 2-ethyl hexoate (POLYCAT 102)
30	0-10	2,2'-oxybis(N,N-dimethylethanamine) (CAS No.- 3033-62-3) (ZF-20)
	0-10	Triethylamine (TEA)
	0-10	Aminified sugar
	0-10	Carboxymethyl cellulose/polyacrylic acid aqueous solution
35	0-10	Aqueous solution of carboxymethyl cellulose
	0-10	Tripropylamine (TPA)
	0-10	Colloidal Silica (SNOWTEX)
	0-1	Antimicrobial/Antibacterial agent

Hard Foam

Hard foams of the present invention are mostly closed cell (about 85% to about 95 %) and have a density from about 1 to about 10 pounds per cubic foot; however, the density of the hard foam can be greater or less than this range. The following side A solution and side B solution, respectively, are admixed in separate mixing vessels, and thereafter mixed with one another in the amounts by weight as indicated above to form a polyurethane hard foam.

## Side A Components:

	Wt. %	Component
	50-100	4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)
	0-50	1-vinyl-2-pyrrolidinone (NVP)
5	0-50	Dimethyl Sulfoxide (DMSO)
	0-20	Silicon surfactant
	0-50	FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

## Side B Components:

	Wt. %	Component
10	0-90	HFP polyester resin/PEG400 blend (50:50 by weight)
	0-90	HFP polyester resin/HEMA blend (50:50 by weight)
	0-50	Vinyl ester resin, resin, or a combination thereof
	0-70	Blend of 25 wt. % HFP polyester resin and 75 wt. % HEMA
15	40-90	Castor Oil
	0-40	Linseed Oil
	0-50	Mineral Oil (heavy mineral oil CAS #8012-95-1, light mineral oil CAS #8042-47-5, or a combination thereof)
	0-50	Glycerol
20	0-30	Polyethylene glycol/dodecylbenzenesulfonic acid solution
	0-50	Ethylene Glycol (EG)
	0-50	A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO <sub>2</sub> (20-21 wt. %) (EG-ST)
	0-50	Polyethylene Glycol 200 molecular weight (PEG200)
25	0-50	Polyethylene Glycol 400 molecular weight (PEG400)
	0-50	Polyethylene Glycol 600 molecular weight (PEG600)
	0-30	FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
	0-30	Triethanolamine (TEOA)
	0-10	Silicon surfactant
30	0-30	2-hydroxyethylmethacrylate (HEMA)
	0-10	4,4-methylenebis(N,N-dimethylaniline) (10 vol. %) in maleate resin (90 vol. %) (AK10)
	0-20	40 vol. % of dibenzyl peroxide in water
	0-30	Calcium oxide (CaO)
35	0-20	Aluminum trihydrate (ATH)
	0-50	CaCO <sub>3</sub>
	0-2	N,N-dimethylaniline (N,N DMA)
	0-2	Cobalt naphthanate (NAPH)
	0-4	Dibutyltin dilaurate (Di-10)
40	0-4	Dipropylene glycol (33LV Catalyst)
	0-10	Bis(dimethyl amino propyl)methyl amine (POLYCAT 77)
	0-10	1,8-diazabicyclo(5,4,0)-7undecane 2-ethyl hexoate (POLYCAT 102)
	0-10	2,2'-oxybis(N,N-dimethylethanamine) (CAS No.- 3033-62-3) (ZF-20)
	0-10	Triethylamine (TEA)
45	0-10	Aminified sugar
	0-10	Water
	0-10	Carboxymethyl cellulose/polyacrylic acid aqueous solution

0-10	Aqueous solution of carboxymethyl cellulose
0-10	Tripropylamine (TPA)
0-10	Colloidal Silica (SNOWTEX)
0-1	Antibacterial/Antimicrobial agent

5

Adhesive Foam

Adhesive foams of the present invention are mostly closed cell (about 85% to about 95 %) and have a density from about 1 to about 4 pounds per cubic foot; however, the density of the adhesive foam can be greater or less than this range. The following side A solution and side B solution, respectively, are admixed in separate mixing vessels, and thereafter mixed with one another in the amounts by weight as indicated above to form a polyurethane adhesive foam. This foam will adhere to wet or dry surfaces or substrates, such as EPDM, concrete, cardboard, plywood, sheetrock, and the like.

## Side A Components:

15	<u>Wt. %</u>	<u>Component</u>
	50-100	4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)
	0-50	1-vinyl-2-pyrrolidinone (NVP)
	0-50	Dimethyl Sulfoxide (DMSO)
	0-20	Silicon surfactant
20	0-50	FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

## Side B Components:

	<u>Wt. %</u>	<u>Component</u>
	0-60	Blend of HFP polyester resin, polyethylene glycol 200, and HEMA
25	0-60	Blend of HFP polyester resin, polyethylene glycol 400, and HEMA
	0-60	Blend of HFP polyester resin, polyethylene glycol 600, and HEMA
	0-90	HFP polyester resin/PEG400 blend (50:50 by weight)
	0-90	HFP polyester resin/HEMA blend (50:50 by weight)
	0-50	Vinyl ester resin, polyester resin, or a combination thereof
30	0-70	Blend of 25 wt. % HFP polyester resin and 75 wt. % HEMA
	40-90	Castor Oil
	0-40	Linseed Oil
	0-50	Mineral Oil (heavy mineral oil CAS #8012-95-1, light mineral oil CAS #8042-47-5, or a combination thereof)
35	0-50	Glycerol
	0-30	Polyethylene glycol/dodecylbenzenesulfonic acid solution
	0-50	Ethylene Glycol (EG)
	0-50	A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO <sub>2</sub> (20-21 wt. %) (EG-ST)
40	0-50	Polyethylene Glycol 200 molecular weight (PEG200)
	0-50	Polyethylene Glycol 400 molecular weight (PEG400)
	0-50	Polyethylene Glycol 600 molecular weight (PEG600)
	0-30	FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

	0-30	Triethanolamine (TEOA)
	0-10	Silicon surfactant
	0-30	2-hydroxyethylmethacrylate (HEMA)
5	0-10	4,4-methylenebis(N,N-dimethylaniline) (10 vol. %) in maleate resin (90 vol. %) (AK10)
	0-20	40 vol. % of dibenzyl peroxide in water
	0-30	Calcium oxide (CaO)
	0-20	Aluminum trihydrate (ATH)
	0-50	CaCO <sub>3</sub>
10	0-10	Fumed Silica
	0-2	N,N-dimethylaniline (N,N DMA)
	0-2	Cobalt naphthanate (NAPH)
	0-4	Dibutyltin dilaurate (Di-10)
	0-10	Dipropylene glycol (33LV Catalyst)
15	0-10	Bis(dimethyl amino propyl)methyl amine (POLYCAT 77)
	0-10	1,8-diazabicyclo(5,4,0)-7undecane 2-ethyl hexoate (POLYCAT 102)
	0-10	2,2'-oxybis(N,N-dimethylethanamine) (CAS No.- 3033-62-3) (ZF-20)
	0-10	Triethylamine (TEA)
	0-10	Aminified sugar
20	0-10	Water
	0-10	Carboxymethyl cellulose/polyacrylic acid aqueous solution
	0-10	Aqueous solution of carboxymethyl cellulose
	0-10	Tripropylamine (TPA)
	0-10	Colloidal Silica (SNOWTEX)
25	0-1	Antimicrobial/Antibacterial agent

This invention is further illustrated by the following examples, which are not to be construed in any way as imposing limitations upon the scope thereof. On the contrary, it is to be clearly understood that resort may be had to various other embodiments, modifications, and equivalents thereof which, after reading the description herein, may suggest themselves to those skilled in the art without departing from the spirit of the present invention. For example, it is to be understood that the amounts of reagents used in the following examples are approximate and that those skilled in the art might vary these amounts and ratios without departing from the spirit of the present invention.

### 35 Examples

In the following examples, unless indicated otherwise, the "A" and "B" side components were prepared at room temperature. Further, there is no requirement for the respective compounds of the "A" and "B" side components to be added and mixed in the order listed.

Example 1

A polyurethane foam was prepared by admixing the following components:

## Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	10 g
dimethyl sulfoxide (DMSO)	4 g

- MDI was added to a mixing vessel (1000ml tri-cornered polypropelene beaker VWR Scientific Cat. 25384-160). DMSO was added to the MDI and mixed thoroughly using a Hamilton Beach Commercial Mixer, Model 95036-3 speeds [low, medium, high] at medium speed for about 5 minutes.

## Side B Components:

Glucose	20 g
Lactose solution	3 g
Aqueous Ammonia	2 g
Acetic Acid	4 g
Dibutyltin dilaurate	0.5 g

- Lactose solution was prepared by mixing lactose in water on a 3:1 ratio by volume. Alternatively, the lactose solution may be obtained commercially with a water content of up to 30% by volume. Aqueous ammonia is a solution of 10% by volume  $\text{NH}_3$  in water.

- Glucose and lactose solution were added to a mixing vessel (1000ml tri-cornered polypropelene beaker VWR Scientific Cat. 25384-160) and mixed thoroughly in the mixer at medium speed for about 5 minutes. Aqueous ammonia was added to the solution and mixed thoroughly in the mixer at medium speed for about 10 minutes. Acetic acid was then added to the solution and mixed thoroughly for about 5 minutes. Next, dibutyltin dilaurate was added to the solution and mixed thoroughly in the mixer at medium speed.

- Following the preparation of the A side and the B side, the two components were mixed together in the mixer at high speed for about 15 seconds. The reaction of side A

with side B resulted in the immediate formation (reaction time of about 5 secs.) of foam cells, which were allowed to freely rise external to the container (rise time about 10 secs.).

The foam cells bonded to all surfaces of the container, and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam was hard and possessed good adherence properties suitable for foam insulating applications to substrates, such as roofing membranes and roofing and wall insulation foams.

#### Example 2a

A polyurethane foam was prepared by admixing the following components in the order as listed:

#### 10 Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)

#### Side B Components:

Castor oil	10 g
Vinyl ester resin (McWhorter Verimac 755-8590)	10 g
Calcium carbonate (CaCO <sub>3</sub> )	20 g
Solution 4	1 g
Dibutyltin dilaurate	0.5 g
Polyethylene glycol-400/dimethacrylate (PEG400/DMA)	1 g
N'N-dimethylaniline (N'N-DMA)	1 g
Cobalt naphthanate	0.5 g
Triethylamine	1 g

Solution 4 (Sol 4) is a mixture employed as a surfactant and comprises:

Carboxymethyl cellulose (CMC)	0.5 g
Polyacrylic acid	0.5 g
Water (H <sub>2</sub> O)	100 ml

The above mixture was thoroughly mixed using a blender for about ten minutes to form the side B component. 15 grams of 4,4'-diphenyl methane diisocyanate (MDI) (sold under the tradename LUPRANATE™) was added to the side B component.

The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time of about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs.). The foam cells bonded to all surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured.

- 5 The resulting foam was hard and possessed good adherence properties, suitable for foam insulating applications.

LUPRANATE™ is the tradename for polymeric diphenyl methane diisocyanate CAS 101-68-8, commonly known as MDI or PMDI, by BASF. Polymeric MDI is a brownish liquid with a functionality between 2.1-3.0.

10 Example 2b

Example 2b was prepared exactly as Example 2a, with the exception that 10 g of unsaturated polyester resin was substituted for vinyl ester resin in the side B component. 15 grams of 4,4'-diphenyl methane diisocyanate (MDI) (LUPRANATE™) was added to the side B component. The reaction of side A with side B resulted in the immediate formation of 15 foam cells (reaction time of about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs.). The foam cells bonded to all surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam was hard and possessed good adherence properties, suitable for foam insulating applications.

20 Example 3a

A polyurethane foam was prepared by admixing the following components in the order as listed:

Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)

25 Side B Components:

Castor oil	100 g
Calcium oxide (CaO)	20 g
Vinyl ester resin	25 g
Calcium carbonate (CaCO <sub>3</sub> )	75 g
Solution 4	2 g



Benzoyl peroxide 20% (BPO in Aqueous solution)	5 g
Dibutyltin dilaurate	1 g
Triethylamine	1 g
2-hydroxyethylmethacrylate	3 g

The above mixture was thoroughly mixed in the mixer at medium speed in a blender for about ten minutes to form the side B component. To this mixture was added 25 grams of 4,4'-diphenyl methane diisocyanate (MDI). The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time about 5 secs.), which were  
5 allowed to freely rise external to the container (rise time about 10 secs.). The foam cells bonded to all surfaces of the container, and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam was hard and possessed good adherence properties, suitable for foam insulating applications.

#### Example 3b

10 Example 3b was prepared exactly as Example 3a, with the exception that 25 g of unsaturated polyester resin was substituted for vinyl ester resin in the side B component. 25 grams of 4,4'-diphenyl methane diisocyanate (MDI) (LUPRANATE™) was added to the side B component. The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time of about 5 secs.), which were allowed to freely rise external to the  
15 container (rise time about 10 secs.). The foam cells bonded to all surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam was hard and possessed good adherence properties, suitable for foam insulating applications.

#### Example 4a

20 A polyurethane foam was prepared by admixing the following components in the order as listed:

Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)

## Side B Components:

Castor oil	20 g
Vinyl ester resin	20 g
Calcium carbonate (CaCO <sub>3</sub> )	63 g
Solution 4	5 g
Dibutyltin dilaurate	1 g

The above mixture was thoroughly mixed in a blender at medium speed for about ten minutes. To this mixture was added 20 grams of 4,4'-diphenyl methane diisocyanate (MDI) (LUPRANATE™). The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs). The foam cells bonded to all surfaces of the container, and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam possessed good adherence properties, suitable for foam insulating applications.

10 Example 4b

Example 4b was prepared exactly as Example 4a, with the exception that 20 g of unsaturated polyester resin was substituted for vinyl ester resin in the side B component. 20 grams of 4,4'-diphenyl methane diisocyanate (MDI) (LUPRANATE™) was added to the side B component. The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time of about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs.). The foam cells bonded to all surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam was hard and possessed good adherence properties, suitable for foam insulating applications.

20

Example 5a

A polyurethane foam was prepared by admixing the following components in the order as listed:

## Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)

## Side B Components:

Castor oil	10 g
Vinyl ester resin	10 g
Calcium carbonate (CaCO <sub>3</sub> )	30 g
Solution 4	1 g
Dibutyltin dilaurate	1 g

5           The above mixture was thoroughly mixed in a blender at medium speed for about ten minutes. To this mixture was added 13 grams of 4,4'-diphenyl methane diisocyanate (MDI) (LUPRANATE™). The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs.). The foam cells bonded to all  
10 surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam was hard and possessed good adherence properties, suitable for foam insulating applications.

Example 5b

15           Example 5b was prepared exactly as Example 5a, with the exception that 10 g of unsaturated polyester resin was substituted for vinyl ester resin in the side B component. 13 grams of 4,4'-diphenyl methane diisocyanate (MDI) (LUPRANATE™) was added to the side B component. The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time of about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs.). The foam cells bonded to all surfaces of the container  
20 and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam was hard and possessed good adherence properties, suitable for foam insulating applications.

Example 6a

A polyurethane foam was prepared by admixing the following components in the order as listed:

Side A Components:

- 5                    4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)

Side B Components:

Castor oil	20 g
Vinyl ester resin	30 g
Calcium carbonate (CaCO <sub>3</sub> )	30 g
Solution 4	5 g
Dibutyltin dilaurate	1 g

The above mixture was thoroughly mixed in a blender at medium speed for about ten minutes. To this mixture was added 16.5 grams of 4,4'-diphenyl methane diisocyanate (MDI) (LUPRANATE™). The reaction of side A with side B resulted in the  
10 immediate formation of foam cells (reaction time about 5 secs.), which were allowed to freely rise external to the container (rise time 10secs). The foam cells bonded to all surfaces of the container, and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam was hard and possessed good adherence properties, suitable for foam insulating applications.

15    Example 6b

Example 6b was prepared exactly as Example 6a, with the exception that 30 g of unsaturated polyester resin was substituted for vinyl ester resin in the side B component. 16.5 grams of 4,4'-diphenyl methane diisocyanate (MDI) (LUPRANATE™) was added to the side B component. The reaction of side A with side B resulted in the immediate  
20 formation of foam cells (reaction time of about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs.). The foam cells bonded to all surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam was hard and possessed good adherence properties, suitable for foam insulating applications.

Example 7a

A polyurethane foam was prepared by admixing the following components in the order as listed:

Side A Components:

- 5                    4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)

Side B Components:

Castor oil	50 g
Vinyl ester resin	50 g
Calcium carbonate (CaCO <sub>3</sub> )	100 g
Triethylamine	2.7 g
Solution 4	2.7 g
Polyethylene glycol-400-dimethacrylate (PEG400/DMA)	2.5 g
Dibutyltin dilaurate	1 g
N,N-dimethylaniline (N,N-DMA)	1 g
Cobalt naphthanate (NAPH)	1 g

- The above mixture was thoroughly mixed in a blender at medium speed for about ten minutes. To this mixture was added 50 grams of 4,4'-diphenyl methane diisocyanate (MDI). The combined mixture was allowed to freely rise. The reaction of side
- 10 A with side B resulted in the immediate formation of foam cells (reaction time about 5 secs.), which were allowed to freely rise external to the container (rise time 10 secs).

Example 7b

- Example 7b was prepared exactly as Example 7a, with the exception that 50 g of unsaturated polyester resin was substituted for vinyl ester resin in the side B component.
- 15 50 grams of 4,4'-diphenyl methane diisocyanate (MDI) (LUPRANATE™) was added to the side B component. The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time of about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs.).

Example 8

A polyurethane foam was prepared by admixing the following components in the order as listed:

## Side A Components:

- 5                    4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)

## Side B Components:

Castor oil	50 g
Calcium oxide (CaO)	10g
Vinyl ester resin	50 g
Calcium carbonate (CaCO <sub>3</sub> )	100 g
Triethylamine	4.2 g
Solution 4	2.7 g
Polyethylene glycol-400-dimethacrylate (PEG400/DMA)	2.5 g
Dibutyltin dilaurate	0.5 g
N'N-dimethylaniline (N'N-DMA)	1 g
Cobalt naphthanate (NAPH)	1 g

- The above mixture was thoroughly mixed in a blender at medium speed for about ten minutes. To this mixture was added 50 grams of 4,4'-diphenyl methane diisocyanate (MDI) (LUPRANATE™). The reaction of side A with side B resulted in the  
10 immediate formation of foam cells (reaction time about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs.).

Example 8b

- Example 8b was prepared exactly as Example 8a, with the exception that 50 g of unsaturated polyester resin was substituted for vinyl ester resin in the side B component.  
15 50 grams of 4,4'-diphenyl methane diisocyanate (MDI) (LUPRANATE™) was added to the side B component. The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time of about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs.).

Example 9

## Preparation of KT Surfactant KT-1S:

KT Surfactant KT-1S comprises a 50:50 by weight mixture of MIX A and MIX B. MIX A comprises a mixture of 50% Polyalkyleneoxide/methylsiloxane Copolymer [CAS 67762-85-0] and 50% Polyalkylene Oxide [CAS 52232-27-6]. Material is sold commercially by Witco Corporation under the trade name of Nix Silicone L 5340. MIX B is a silicon glycol polymer in liquid form sold under the trade name Dow Corning 193 Surfactant. Dow Corning 193 Surfactant is a copolymer that acts as a surface tension depressant and wetting agent. The composition contains dimethyl, methyl (polythene oxide) siloxane, polyethylene oxide monoallyl ether, and polyethylene glycol.

## Preparation of KT Initiator KT1I :

A polyaniline was prepared according to the following protocol:

A 500ml solution of 1Mol hydrochloric acid (HCl) [CAS 7467-01-0] /H<sub>2</sub>O is made up ( $12 \times 500 \times 1 \text{ 500} / 12 = 42 \text{ ml HCl} / 458 \text{ ml H}_2\text{O}$ ). Of this 500ml HCl aqueous solution, 200ml is measured and poured into an Erlenmeyer flask; 12 grams of ammonium-persulfate (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> is then added to the 200ml solution. The solution is stirred until the ammonium-persulfate is completely dissolved. The solution is then placed in an ice bath (0° C) to control the reaction, which could be exothermic.

The remaining HCl/aqueous solution is transferred to a reaction flask and placed in an ice bath at 3° C. The temperature of the solution is allowed to rise to 5° C by the exotherm of the solution. 21ml of distilled aniline (ref: Aldrich cat. 24,228-4 [CAS 62-53-3]) is measured and added to the reaction flask. The solution is placed on a magnetic stir bar and stirred until completely mixed.

The prepared solution of HCl/ammonium-persulfate is added to the reaction flask and stirred until the mixture is completely homogenized; the temperature of the ice bath is reduced to 0° C to control the reaction, which could be exothermic for approximately 20 minutes. The mixed polyaniline solution is then vacuum filtered by placing the solution on filter paper in a vacuum funnel. The funnel is placed on an Erlenmeyer flask equipped with a side arm. Vacuum is then pulled on the solution leaving the residue on the filter paper.

The vacuum filtered polyaniline is transferred into a flask containing 1Mol potassium hydroxide (KOH) [CAS 1310-58-3] (6 grams KOH per 100ml H<sub>2</sub>O). The mixture

is stirred for about 2 hours, then left standing for a period of eight hours to allow the solution to settle.

- 5 The polyaniline is then mixed with 500ml of filtered (deionized) water and mixed until homogenous. The polyaniline is then baked until dried in an oven at 50°C. The final polyaniline powder has the appearance of a black-green powder. The powder is then placed in a sealable bottle and purged with nitrogen until ready for use in the Side B Components.

#### Example 10

A polyurethane foam was prepared by admixing the following components:

#### 10 Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	60 g
1-Vinyl-2-pyrrolidinone (NVP)	10 g
KT Surfactant KT-1S	1 g
FYROL™ (tri (2-chloroethyl)phosphate flame retardant)	10 g

- MDI was added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160). NVP was added to MDI and mixed thoroughly in the mixer at medium speed for about 5 minutes. The KT Surfactant KT-1S was then added into the solution and mixed thoroughly for about 5 minutes. Then, FYROL™ was added into the solution and mixed thoroughly for about 10-15 minutes.

#### Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	50g
Water	10g
KT Initiator KT-1I	0.5g
N'N-dimethylaniline (N'N-DMA)	0.5g

Water and granulated sugar were added to a mixing vessel and mixed thoroughly in a blender at medium speed until the sugar dissolved, about 5 minutes at about



40°C. KT-1I Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly.

Following the preparation of side solutions A and B, the two solutions were mixed together in the mixer at high speed. The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time about 5 secs.), which were allowed to  
5 freely rise external to the container (rise time about 10 secs.).

### Example 11

A polyurethane foam was prepared by admixing the following components:

#### 10 Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	60 g
1-Vinyl-2-pyrrolidinone (NVP)	10 g
KT Surfactant KT-1S	1 g
FYROL™ (tri (2-chloroethyl)phosphate flame retardant)	10 g

A mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160) was purged with nitrogen. MDI was added to the vessel. NVP was added to the LUPRANATE™ and mixed thoroughly in the mixer at high speed for about 5  
15 minutes. The KT-1S Surfactant was then added into the solution and mixed thoroughly for about 5 minutes. Then, FYROL™ was added into the solution and mixed thoroughly for about 10-15 minutes.

#### Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	50g
Water	10g
KT Initiator KT-1I	0.5g
N'N-dimethylaniline (N'N-DMA)	0.5g

The water and granulated sugar were added to a vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160) and mixed thoroughly in a blender at medium speed until the sugar dissolved, about 5 minutes at about 40°C. KT-1I Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly.

Following the preparation of side solutions A and B, the two solutions were mixed together. To the mixed solution, was added 1 gram of triethylamine [CAS 121-44-8]. The triethylamine [TEA] acts as a corebinding agent and accelerator of the reaction. As a corebinding agent, the TEA aides in the construction of inner and outer skin attaching of the cells in the honeycomb of the foam. The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time about 5 secs.), which were allowed to freely rise external to the container (rise time 10secs). The foam cells bonded to all surfaces of the container, and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam possessed good adherence properties, suitable for foam insulating applications.

#### Example 12

A polyurethane foam was prepared by admixing the following components:

Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	50 g
1-Vinyl-2-pyrrolidinone (NVP)	10 g
KT Surfactant KT-1S	1 g
FYROL™ (tri (2-chloroethyl)phosphate flame retardant)	5 g

MDI was added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160). NVP was added to the MDI and mixed thoroughly for about 5 minutes. The KT-1S Surfactant was then added into the solution and mixed thoroughly for about 5 minutes. Then, FYROL™ was added into the solution and mixed thoroughly for about 10-15 minutes.

## Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] $C_{12}H_{22}O_{11}$	50g
Water	10g
KT Initiator KT-1I	1g
N,N-dimethylaniline (N,N-DMA)	0.5g

The water and granulated sugar were added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160) and mixed thoroughly until the sugar dissolved, about 5 minutes at about 40°C. KT-1I Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly.

Following the preparation of side solutions A and B, the two solutions were mixed together. To the mixed solution was added 2 grams of triethylamine [CAS 121-44-8]. The triethylamine acts as a corebinding agent. The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs.). The foam cells bonded showed a high heat evolution.

Example 13

A polyurethane foam was prepared by admixing the following components:

## Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	50 g
1-Vinyl-2-pyrrolidinone (NVP)	5 g
KT Surfactant KT-1S	1 g

MDI was added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160). NVP was added to the MDI and mixed thoroughly for about 5 minutes. The KT-1S Surfactant was then added into the solution and mixed thoroughly for about 5 minutes.

## Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	50g
Water	10g
KT Initiator KT-11	1g
N,N-dimethylaniline (N,N-DMA)	1g

The water and granulated sugar were added to a mixing vessel (1000ml tri-cornered polypropelene beaker VWR Scientific Cat. 25384-160) and mixed thoroughly until the sugar dissolved, about 5 minutes at about 40°C. KT-11 Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly.

Following the preparation of side solutions A and B, the two solutions were mixed together. To the mixed solution was added 1 gram of triethylamine [CAS 121-44-8]. The triethylamine acts as a corebinding agent. The reaction of side solution A and side solution B resulted in the formation of foam cells, which were allowed to freely rise.

Example 14

A polyurethane foam was prepared by admixing the following components:

## Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	50 g
1-Vinyl-2-pyrrolidinone (NVP)	5 g
KT Surfactant KT-1S	1 g

MDI was added to a mixing vessel (1000ml tri-cornered polypropelene beaker VWR Scientific Cat. 25384-160). NVP was added to the MDI and mixed thoroughly for about 5 minutes. The KT-1S Surfactant was then added into the solution and mixed thoroughly for about 5 minutes.

## Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	50g
Water	10g
KT Initiator KT-1I	0.25g
N,N-dimethylaniline (N,N-DMA)	0.1g

The water and granulated sugar were added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160) and mixed thoroughly until the sugar dissolved, about 5 minutes at about 40°C. KT-1I Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly.

Following the preparation of side solutions A and B, the two solutions were mixed together. To the mixed solution was added 1 gram of triethylamine [CAS 121-44-8]. The triethylamine acts as a corebinding agent. The reaction of side solution A and side solution B resulted in the formation of foam cells, which were allowed to freely rise. The cellular structure was consistent; however, upon final cure it was noted that shrinkage of about 10% of original volume had occurred.

Example 15

A polyurethane foam was prepared by admixing the following components:

## 15 Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	40 g
1-Vinyl-2-pyrrolidinone (NVP)	7 g
KT Surfactant KT-1S	1 g
FYROL™ (tri (2-chloroethyl)phosphate flame retardant)	5 g

MDI was added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160). NVP was added to the MDI and mixed thoroughly for about 5 minutes. The KT-1S Surfactant was then added into the solution and mixed thoroughly for about 5 minutes. Then, FYROL™ was added into the solution and mixed thoroughly for about 10-15 minutes.

Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	20g
Water	10g
KT Initiator KT-1I	0.35g
N,N-dimethylaniline (N,N-DMA)	0.35g

The water and granulated sugar were added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160) and mixed thoroughly until the sugar dissolved, about 5 minutes at about 40°C. KT-1I Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly.

Following the preparation of side solutions A and B, the two solutions were mixed together. To the mixed solution was added 1 gram of triethylamine [CAS 121-44-8]. The triethylamine acts as a corebinding agent. The reaction of side solution A and side solution B resulted in the formation of foam cells, which were allowed to freely rise. The foam cells bonded to all surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam possessed good adherence properties, suitable for foam insulating applications.

#### Example 16

A polyurethane foam was prepared by admixing the following components:

Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	45 g
1-Vinyl-2-pyrrolidinone (NVP)	7 g

KT Surfactant KT-1S	1 g
FYROL™ (tri (2-chloroethyl)phosphate flame retardant)	5 g

MDI was added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160). NVP was added to the MDI and mixed thoroughly for about 5 minutes. The KT-1S Surfactant was then added into the solution and mixed thoroughly for about 5 minutes. Then, FYROL™ was added into the solution and mixed  
5 thoroughly for about 10-15 minutes. Crosslinking agent PTT was added to the A side following which the solution was thoroughly mixed for an additional 10 minutes.

Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	20g
Water	10g
KT Initiator KT-1I	0.35g
N,N-dimethylaniline (N,N-DMA)	0.35g
Silica (silicon dioxide, SiO <sub>2</sub> )	20g
Pentaerythritol tetraacrylate (PTT)	5g

The water and granulated sugar were added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160) and mixed thoroughly until  
10 the sugar dissolved, about 5 minutes at about 40°C. KT-1I Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly. Silica was then added to the solution and thoroughly mixed.

Following the preparation of side solutions A and B, the two solutions were  
15 mixed together. To the mixed solution was added a mixture of 1 gram of triethylamine [CAS 121-44-8] and 1 gram chloroform. The reaction of side solution A and side solution B resulted in the formation of foam cells, which were allowed to freely rise. The foam cells

bonded to all surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam possessed good adherence properties, suitable for foam insulating applications.

### Example 17

5 A polyurethane foam was prepared by admixing the following components:

Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	45 g
1-Vinyl-2-pyrrolidinone (NVP)	7 g
KT Surfactant KT-1S	1 g
FYROL™ (tri (2-chloroethyl)phosphate flame retardant)	5 g

MDI was added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160). NVP was added to the MDI and mixed thoroughly for about 5 minutes. The KT-1S Surfactant was then added into the solution and mixed  
10 thoroughly for about 5 minutes. Then, FYROL™ was added into the solution and mixed thoroughly for about 10-15 minutes. Crosslinking agent PTT was added to the A side following which the solution was thoroughly mixed for an additional 10 minutes.

Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	20g
Water	10g
KT Initiator KT-1I	0.35g
N,N-dimethylaniline (N,N-DMA)	0.35g
Silica (silicon dioxide SiO <sub>2</sub> )	20g
Pentaerythritol tetraacrylate (PTT)	5g



The water and granulated sugar were added to the mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160) and mixed thoroughly until the sugar dissolved, about 5 minutes at about 40°C. KT-1I Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly. Silica was then added to the solution and thoroughly mixed.

Following the preparation of side solutions A and B, the two solutions were mixed together. To the mixed solution was added a mixture of 1 gram of triethylamine [CAS 121-44-8] and 1 gram chloroform [CAS 67-66-3]. The reaction of side solution A and side solution B resulted in the formation of foam cells, which were allowed to freely rise. The foam cells bonded to all surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam possessed good adherence properties, suitable for foam insulating applications.

#### Example 18

A polyurethane foam was prepared by admixing the following components:

#### Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	45 g
1-Vinyl-2-pyrrolidinone (NVP)	7 g
KT Surfactant KT-1S	1 g
FYROL™ (tri (2-chloroethyl)phosphate flame retardant)	5 g

MDI was added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160). NVP was added to the MDI and mixed thoroughly for about 5 minutes. The KT-1S Surfactant was then added into the solution and mixed thoroughly for about 5 minutes. Then, FYROL™ was added into the solution and mixed thoroughly for about 10-15 minutes. Crosslinking agent PTT was added to the A side following which the solution was thoroughly mixed for an additional 10 minutes.

## Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	20g
Water	10g
KT Initiator KT-1I	0.35g
N,N-dimethylaniline (N,N-DMA)	0.35g
Pentaerythritol tetraacrylate (PTT)	5g

The water and granulated sugar were added to a mixing vessel (1000ml tri-cornered polypropylene beaker VWR Scientific Cat. 25384-160) and mixed thoroughly until the sugar dissolved, about 5 minutes at about 40°C. KT-1I Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly.

Following the preparation of side solutions A and B, the two solutions were mixed together. To the mixed solution was added a mixture of 1 gram of triethylamine [CAS 121-44-8] and 1 gram chloroform [CAS 67-66-3]. The triethylamine/ chloroform mix acts as a corebinding agent and as an additional blowing agent. The reaction of side A and side B resulted in the formation of foam cells, which were allowed to freely rise. The foam cells bonded extremely well to all surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam possessed good adherence properties, suitable for foam insulating applications.

15 Example 19

A polyurethane foam was prepared by admixing the following components:

## Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	2,250 g
1-Vinyl-2-pyrrolidinone (NVP)	350 g
KT Surfactant KT-1S	50 g

FYROL™ (tri (2-chloroethyl)phosphate flame retardant)	250 g
---	-------

MDI was added to a vessel. NVP was added to the MDI and mixed thoroughly for about 5 minutes. The KT-1S Surfactant was then added into the solution and mixed thoroughly for about 5 minutes. Then, FYROL™ was added into the solution and mixed thoroughly for about 10-15 minutes. Crosslinking agent PTT was added to the A side following which the solution was thoroughly mixed for an additional 10 minutes.

Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	1,000g
Water	500g
KT Initiator KT-1I	17.5g
N,N-dimethylaniline (N,N-DMA)	17.5g
Pentaerythritol tetraacrylate (PTT)	250g

The water and granulated sugar were added to a vessel and mixed thoroughly until the sugar dissolved, about 5 minutes at about 40°C. KT-1I Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly.

Following the preparation of side solutions A and B, the two solutions were mixed together. To the mixed solution was added a mixture of 50 grams of triethylamine [CAS 121-44-8] and 50 grams chloroform [CAS 67-66-3]. The triethylamine/ chloroform mix acts as a corebinding agent and as an additional blowing agent. The reaction of side A and side B resulted in the formation of foam cells, which were allowed to freely rise. The foam cells bonded extremely well to all surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam possessed good adherence properties, suitable for foam insulating applications.

#### Example 20

A polyurethane foam was prepared by admixing the following components:

## Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	5,750 g
1-Vinyl-2-pyrrolidinone (NVP)	1,050 g
KT Surfactant KT-1S	150 g
FYROL™ (tri (2-chloroethyl)phosphate flame retardant)	750 g

MDI was added to a vessel. NVP was added to the MDI and mixed thoroughly for about 5 minutes. The KT-1S Surfactant was then added into the solution and mixed thoroughly for about 5 minutes. Then, FYROL™ was added into the solution and mixed thoroughly for about 10-15 minutes. Crosslinking agent PTT was added to the A side following which the solution was thoroughly mixed for an additional 10 minutes.

## Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	3,000g
Water	1,500g
KT Initiator KT-1I	52.5g
N,N-dimethylaniline (N,N-DMA)	52.5g
Pentaerythritol tetraacrylate (PTT)	250g

The water and granulated sugar were added to a vessel and mixed thoroughly until the sugar dissolved, about 5 minutes at about 40°C. KT-1I Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly.

Following the preparation of side solutions A and B, the two solutions were mixed together. To the mixed solution was added a mixture of 150 grams of triethylamine [CAS 121-44-8] and 150 grams chloroform [CAS 67-66-3]. The triethylamine/ chloroform mix acts as a corebinding agent and as an additional blowing agent. The reaction of side A

and side B resulted in the formation of foam cells, which were allowed to freely rise. The foam cells bonded extremely well to all surfaces of the container and the resultant foam remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam possessed good adherence properties, suitable for foam insulating applications.

## 5 Example 21

Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	350lb
1-Vinyl-2-pyrrolidinone (NVP)	100lb
KT Surfactant KT-1S	10lb
FYROL™ (tri (2-chloroethyl)phosphate flame retardant)	50lb

MDI was added to a mixing vessel. NVP was added to the LUPRANATE™ and mixed thoroughly for about 5 minutes. The KT-1S Surfactant was then added into the solution and mixed thoroughly for about 5 minutes. Then, FYROL™ was added into the solution and mixed thoroughly for about 10-15 minutes. The mixing vessel was fitted with a drum pump [IPM Kent, WA. Model IP-01].

Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	350lb
Water	100lb
KT Initiator KT-1I	10lb
N'N-dimethylaniline (N'N-DMA)	2.5lb

The water and granulated sugar were added to a mixing vessel (Stainless Steel 100, gallon capacity fitted with a drum heater (Acra Electric Corporation Model 152274) and mixed thoroughly using a Neptune Mixer, 1/3HP at 1725 RPM until the sugar dissolved, about 45 minutes at about 80 °C. KT-1I Initiator was added to the solution and mixed

thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution and mixed thoroughly. The mixture was then transferred to a 55 gallon drum fitted with a drum pump (IPM Kent, WA. Model IP-01, 180psi) and a mixer (Neptune Mixer, 1/3HP at 1725 RPM).

5 The drum pumps were connected to standard spray gun (airless urethane spray equipment Gusmer Corporation H-2000 Multi-Component High Pressure Metering Unit with a Gusmer GX-7 Spray Gun). Following the preparation of side solutions A and B, the two solutions were mixed together in the mixing chamber of the spray gun, which mixes the components at or approximately at the nozzle, and sprayed into a mold (a simulated 2 inch x 10 4 inch Stud Wall Cavity). The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time about 5 secs.), which were allowed to freely rise external to the container (rise time 10secs).

A second test was performed by introducing the mixture into a mold by use of a conventional Gusmer GX-14 Pour Gun that mixes side A and side B at or approximately at 15 the nozzle. The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time about 5 secs.), which were allowed to freely rise external to the container (rise time about 10 secs.).

In both tests, the foam cells bonded to all surfaces of the mold and remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam 20 possessed good adherence properties, suitable for foam insulating applications.

#### Example 22

Side A Components:

4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)	454.0lb
KT Surfactant KT 1S	9.5lb

In a mixing vessel, the KT Surfactant KT 1S was added directly into the MDI and mixed thoroughly.

25

## Side B Components:

Granulated sugar (crushed) (SUCROSE) [CAS 57-50-1] C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	189.35 lb
Water	94.67 lb
N-vinyl-2-pyrrolidone (NVP)	75.74 lb
N,N Dimethyl aniline (DMA)	4.98 lb
Dimethyl Sulfoxide (DMSO)	37.87 lb
KT Initiator KT-11	1.49 lb
Triethylamine	4.98 lb
Triethanolamine	28.40lb
Aminified Sugar	14.20 lb
FYROL™ (tri (2-chloroethyl)phosphate flame retardant)	47.33 lb

Preparation of Aminified Sugar (Sol AS):

An aminified sugar solution was prepared by mixing microwave initiator 0.1  
 5 % by weight to diethyl cyclohexylamine 99 % by weight.

Preparation of microwave initiator

A microwave initiator was prepared by the following procedure. A first solution was prepared by mixing the following components, in descending order.

- 10 194.52gr. polyvinylalcohol solution (10% PVOH in H<sub>2</sub>O)  
 48.64gr. saturated solution of polyaniline in N-methyl-pyrrolidinone  
 (polyaniline is dissolved in tetrahydrofuran – THF to make saturated solution)  
 19.47gr. ethylene glycol  
 14.60gr. Copper perchlorate (CuClO<sub>4</sub>) saturated solution in methanol

15 The above solution was placed in an ice bath. To this solution was added 60.77grams of concentrated HCL (hydrochloric acid). This solution is designated M1. Total weight – first solution M1 - 338grams

To the above solution M1 was added an equal amount (ratio 1:1 by weight) of hydrogen peroxide  $H_2O_2$  (30% by weight). 338 grams total weight MI-B in sample 676 grams/1.49lbs.

The Microwave initiator is kept refrigerated until ready for us.

5           The water and granulated sugar were added to a mixing vessel (Stainless Steel 100, gallon capacity fitted with a drum heater (Acra Electric Corporation Model 152274) and mixed thoroughly using a Neptune Mixer, 1/3HP at 1725 RPM until the sugar dissolved, about 45 minutes at about 80° C. KT-1I Initiator was added to the solution and mixed thoroughly for about 10 minutes. Next, the N,N-dimethylaniline was added to the solution  
10 and mixed thoroughly. Finally, the remaining components of the side B solution were added to the mixing vessel and mixed thoroughly. The mixture was then transferred to a 55 gallon drum fitted with a drum pump (IPM Kent, WA. Model IP-01, 180psi) and a mixer (Neptune Mixer, 1/3HP at 1725 RPM). The drum pumps were connected to standard airless urethane spray equipment (Gusmer Corporation H-2000 Multi-Component High Pressure Metering  
15 Unit with a Gusmer GX-7 Spray Gun).

Following the preparation of side solutions A and B, the two solutions were mixed together in the mixing chamber of the GX-7 Spray Gun and sprayed on a simulated 2 inch x 4 inch Stud Wall Cavity. The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time about 5 secs.), which were allowed to freely rise  
20 external to the container (rise time about 10 secs.).

A second test was performed by introducing the mixture into a mold by use of a conventional Gusmer GX-14 Pour Gun that mixes side A and side B at or approximately at the nozzle. The reaction of side A with side B resulted in the immediate formation of foam cells (reaction time about 5 secs.), which were allowed to freely rise external to the container  
25 (rise time 10secs).

In both tests, the foam cells bonded to all surfaces of the mold and remained "tacky" for approximately 15-20 minutes until completely cured. The resulting foam possessed good adherence properties, suitable for foam insulating applications.

Examples 23-77 were prepared as follows unless indicated otherwise. In each  
30 of the following polyurethane foam compositions, the side A and side B components were admixed in separate mixing vessels in the order as listed. However, there is no requirement



for the components to be added to the respective mixing vessels in the order listed. Rather, the side A and side B components can be added to the respective mixing vessels any order. After the elected components were placed into the mixing vessel, the components were thoroughly mixed. After the side A and side B components, respectively, were admixed into solutions, the side A and side B solutions are admixed and allowed to react with one another to form a polyurethane foam. The side A solutions and the side B solutions were mixed 1:1 by weight.

#### INSULATION FOAM (Examples 23-31)

##### 10 Example 23

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

##### Side B Component:

- 15 35.0 g Corn Syrup
- 2.0g 1-vinyl-2-pyrrolidinone (NVP)
- 3.0g Ethylene Glycol (EG)
- 5.0g FYROL™ (tri(2-chloroethyl)phosphate flame retardant)
- 20 3.0g Triethanolamine (TEOA)
- 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicon L5340)
- 0.5g Propoxylated polyoxyethylene (Antarox 25-R-2)
- 0.07g Dibutyltin dilaurate (Di-10)
- 1.0g 2,2'-oxybis(N,N-dimethylethanamine) (CAS No.- 3033-62-3) (ZF-20)

25 The side B solution was mixed with MDI (BASF M20) at 1:1 ratio by weight to give a 2-3 second initiation and 5-6 second rise and cure. This example has a good body and firm and some flexibility.

##### 30 Example 24

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

##### Side B Components:

- 35 35.0 g Corn Syrup
- 2.0g 1-vinyl-2-pyrrolidinone (NVP)
- 3.0g A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO<sub>2</sub> (20-21 wt. %) (EG-ST)
- 5.0g FYROL™ (tri(2-chloroethyl)phosphate flame retardant)

- 3.0g Triethanolamine (TEOA)
- 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicon L5340)
- 0.5g Propoxylated polyoxyethylene (Antarox 25-R-2)
- 0.07g Dibutyltin dilaurate (Di-10)
- 5 1.0g 2,2'-oxybis(N,N-dimethylethanamine) (CAS No.- 3033-62-3) (ZF-20)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 2-3 second initiation and 5-6 second rise and cure. This example had a good body and firm and some flexibility.

10

#### Example 25

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- 15 Side B components:
- 35.0 g Corn Syrup
- 2.0g 1-vinyl-2-pyrrolidinone (NVP)
- 3.0g Polyethylene Glycol 200 (PEG200)
- 5.0g FYROL™ (tri(2-chloroethyl)phosphate flame retardant)
- 20 3.0g triethanolamine (TEOA)
- 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicon L5340)
- 0.5g Propoxylated polyoxyethylene (Antarox 25-R-2)
- 0.07g Dibutyltin dilaurate (Di-10)
- 1.5g 2,2'-oxybis(N,N-dimethylethanamine) (CAS No.- 3033-62-3) (ZF-20)

25

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 2-3 second initiation and 3-4 second rise and cure. This sample had more firm body and faster initiation and cure.

#### 30 Example 26a

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B Components:

- 35.0 g Corn Syrup
- 35 2.0g 1-vinyl-2-pyrrolidinone (NVP)
- 3.0g Polyethylene Glycol 400 (PEG400)
- 5.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
- 3.0g Triethanolamine (TEOA)
- 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicon L5340)
- 40 0.5g Propoxylated polyoxyethylene (Antarox 25-R-2)
- 1.5g 2,2'-oxybis(N,N-dimethylethanamine) (CAS No.- 3033-62-3) (ZF-20)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 2-3 second initiation and 3-4 second rise and cure. This sample has more firm body and faster initiation and cure.

5

#### Example 26b

This example was prepared exactly as Example 26a with the exception that the side B solution included 0.1 grams of the Antimicrobial/Antibacterial agent. This is an antimicrobial/antibacterial composition employed to prevent fungus or bacterial growth sold under the trade name INTERCEPT 100% ACTIVE.

10

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 2-3 second initiation and 3-4 second rise and cure. This sample has more firm body and faster initiation and cure.

#### 15 Example 27

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

##### Side B Components:

- 30.0g Sugar Syrup
- 20 7.0g 1-vinyl-2-pyrrolidinone (NVP)
- 0.25g N'N-dimethylaniline (N'N DMA)
- 3.0g Dimethyl Sulfoxide (DMSO)
- 5.0g FYROL™ (tri(2-chloroethyl)phosphate flame retardant)
- 3.0g Triethanolamine (TEOA)
- 25 0.5g Triethylamine (TEA)
- 1.5g Aminified sugar

The side B solution was mixed with MDI /WS at 1:1 ratio by weight to give a 4-5 second initiation and 6-7 second rise and cure. This sample had firm body and slower initiation and cure, and shrunk after cure.

30

MDI/WS was prepared by thoroughly mixing the following at room temperature:  
100.0g 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI)  
1.0g Propoxylated polyoxyethylene (Antarox 25-R-2)  
35 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

Example 28

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

## Side B Components:

- 5 33.0g Sugar Syrup
- 4.0g 1-vinyl-2-pyrrolidinone (NVP)
- 0.25g N'N-dimethylaniline (N'N DMA)
- 3.0g Ethylene Glycol (EG)
- 5.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
- 10 3.0g Triethanolamine (TEOA)
- 1.0g Propoxylated polyoxyethylene (Antarox 25-R-2)
- 0.5g Triethylamine (TEA)
- 1.5g Aminified sugar

- 15 The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 4-5 second initiation and 6-7 second rise and cure. This sample had firm body and slower initiation and cure, and was more flexible.

20 Example 29a

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

## Side B Components:

- 33.0g Sugar Syrup
- 25 4.0g 1-vinyl-2-pyrrolidinone (NVP)
- 0.25g N'N-dimethylaniline (N'N DMA)
- 3.0g Ethylene Glycol (EG)
- 5.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
- 3.0g Triethanolamine (TEOA)
- 30 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 0.25g Dibutyltin dilaurate (Di-10)
- 0.5g Triethylamine (TEA)
- 1.5g Aminified Sugar (Sol AS)

- 35 The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 4-5 second initiation and 6-7 second rise and cure. This sample had firm body and faster initiation and cure, was more flexible, and had better cell structure.

Example 29b

This example was prepared exactly as Example 26a with the exception that the side B solution included 0.2 grams of the Antimicrobial/Antibacterial agent.

5           The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 4-5 second initiation and 6-7 second rise and cure. This sample has more firm body and faster initiation and cure.

10   Example 30

Preparation of Polyol #1:

15       Polyol #1 was formed by mixing about 200g of Corn Syrup with about 6g of sucrose at about 40° C. until the sucrose dissolved. In the present invention, the ratio of sucrose to corn syrup can be varied up to 1:1 by weight.

Example 30 is the same as Example 23, except polyol #1 is substituted for corn syrup.

20       In this example a better cell structure is observed and a more firmed body to the foam with respect to Example 23.

Example 31

25       The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B Components:

25.0g Sugar Syrup (2:1) (a solution comprising 2 parts sugar or sucrose to 1 part water by weight)  
25.0g Polyethylene Glycol 600 (PEG600)  
30   2.0g Sol 4 (see Example 2)  
0.5g Dibutyltin dilaurate (Di-10)  
0.5g Tripropylamine (TPA)  
1.5g Aminified sugar

35       The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 4-5 second initiation and 6-7 second rise and cure. This foam was more flexible and had a softer texture.

## ROOFING FOAM (Examples 32-35)

The following mixes were prepared by thoroughly mixing the listed components in order at room temperature.

5

Mix #1

300.0g Castor Oil

10.0g Ethylene Glycol (EG)

10 10.0g Light mineral oil (LMO)

10.0g Triethanolamine (TEOA)

20.0g Glycerol

20.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

10.0g Colloidal Silica (SNOWTEX)

15

Mix #2

300.0g Castor Oil

10.0g Polyethylene Glycol 400 (PEG400)

20 10.0g Light mineral oil (LMO)

10.0g Triethanolamine (TEOA)

20.0g Glycerol

20.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

10.0g Colloidal Silica (SNOWTEX)

25

Mix #3

300.0g Castor Oil

30 10.0g A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO<sub>2</sub> (20-21 wt. %) (EG-ST)

10.0g Heavy mineral oil (HMO)

10.0g Triethanolamine (TEOA)

20.0g Glycerol

20.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

35 10.0g Colloidal Silica (SNOWTEX)

Mix #4

300.0g Castor Oil

40 10.0g Mix #5 (see below)

10.0g Heavy mineral oil (HMO)

10.0g Triethanolamine (TEOA)

20.0g Glycerol

20.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

45 10.0g Colloidal Silica (SNOWTEX)

Mix #5

The following components were thoroughly mixed at about 40° C until the dodecylbenzenesulfonic acid dissolved.

5 150.0g Polyethylene Glycol 200 (PEG200) (or PEG400 or PEG600)

5.0g Dodecylbenzenesulfonic Acid, sodium salt form (DBSA)

In the present invention, the amount of DBSA can be increased up to about 25% by weight of Mix #5.

10 Example 32

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

50.0g mix #1

15 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

0.3g Dibutyltin dilaurate (Di-10)

0.8g Dipropylene glycol (33LV Catalyst)

20 The side B solution was mixed for 10 seconds with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 1-2 second rise and cure. The foam was hard with some flex to it.

Example 33

25 The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

50.0g mix #2

1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

0.3g Dibutyltin dilaurate (Di-10)

30 0.8g Dipropylene glycol (33LV Catalyst)

The side B solution was mixed for 10 seconds with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 1-2 second rise and cure. The foam feels harder yet with some flex and better cell structure.

35

Example 34

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

40

## Side B components:

- 50.0g mix #3  
1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)  
0.3g Dibutyltin dilaurate (Di-10)  
5 0.8g Dipropylene glycol (33LV Catalyst)

The side B solution was mixed for 10 seconds with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 1-2 second rise and cure. The foam feels harder yet with some flex and better cell structure.

10

Example 35

- The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

## Side B components:

- 50.0g mix #4  
1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)  
0.3g Dibutyltin dilaurate (Di-10)  
20 0.8g Dipropylene glycol (33LV Catalyst)

- The side B solution was mixed for 10 seconds with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to give a 1-2 second rise and cure. The foam felt harder, yet with some flex. Mix #4 it gave a better adhesions foam and cell structure.

## MOLDED FOAM (Examples 36-50)

30 Example 36

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

## Side B components:

- 30.0g Castor oil  
35 3.0g Calcium oxide (CaO)  
5.0g Ethylene Glycol (EG)  
5.0g Aluminum trihydrate (ATH)  
5.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)  
1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)  
40 0.55g Dipropylene glycol (33LV Catalyst)



0.015g Dibutyltin dilaurate (Di-10)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight. This foam was hard with some flex to it, and the reaction time and cure was fast.

#### Example 37

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- Side B components:
- 70.0g Castor oil
  - 7.0g Calcium oxide (CaO)
  - 10.0g Ethylene Glycol (EG)
  - 10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
  - 2.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
  - 0.3g Dipropylene glycol (33LV Catalyst)
  - 0.025g Dibutyltin dilaurate (Di-10)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight at 45 seconds to initiate, 100 seconds to gel and cure, and 130 seconds to foam and rise. The foam had a good firm body at 6 minutes. This foam was hard with some flex.

#### Example 38

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- Side B components:
- 70.0g Castor oil
  - 7.0g Calcium oxide (CaO)
  - 10.0g Polyethylene Glycol 600 (PEG600)
  - 10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
  - 2.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
  - 0.3g Dipropylene glycol (33LV Catalyst)
  - 0.025g Dibutyltin dilaurate (Di-10)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight at 45 seconds to initiate, 100 seconds to gel and cure, and 130 seconds to foam and rise. The foam had a good firm body at 6 minutes. This foam was hard with some flex.

Example 39

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- 5 Side B components:  
70.0g Castor oil  
7.0g Calcium oxide (CaO)  
10.0g A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO<sub>2</sub> (20-21 wt. %) (EG-ST)  
10 10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)  
2.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)  
0.3g Dipropylene glycol (33LV Catalyst)  
0.025g Dibutyltin dilaurate (Di-10)

- 15 The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight at 45 seconds to initiate, 100 seconds to gel and cure, and 130 seconds to foam and rise. The foam had a good firm body at 6 minutes. This foam was hard with some flex.

20 Example 40

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- Side B components:  
70.0g Castor oil  
25 0.6g Calcium oxide (CaO)  
15.0g Ethylene Glycol (EG)  
10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)  
1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)  
1.0g Dipropylene glycol (33LV Catalyst)  
30 0.025g Dibutyltin dilaurate (Di-10)

- The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight at 45 seconds to initiate, 100 seconds to gel and cure, and 130 seconds to foam and rise. The foam had a good firm body at 6 minutes.  
35 This foam was hard with some flex.

Example 41

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- 40 Side B components:

70.0g Castor oil  
0.4g Calcium oxide (CaO)  
15.0g A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO<sub>2</sub> (20-21 wt. %) (EG-ST)  
5 10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)  
1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)  
0.7g Dipropylene glycol (33LV Catalyst)  
0.025g Dibutyltin dilaurate (Di-10)

10 The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight. The reaction time was fast. The foam had a good firm body at 6 minutes. This foam was hard with some flex.

15 Example 42

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

70.0g Castor oil  
20 15.0g Ethylene Glycol (EG)  
10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)  
1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)  
0.4g Dipropylene glycol (33LV Catalyst)  
0.02g Dibutyltin dilaurate (Di-10)  
25 0.4g 1,8-diazabicyclo(5,4,0)-7undecane 2-ethyl hexoate, CAS # 33918-18-2 (Polycat SA102)  
0.3g Bis(dimethyl amino propyl)methyl amine (POLYCAT 77)

30 The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight at 45 seconds to initiate, 100 seconds to gel and cure, and 130 seconds to foam and rise. The foam had a good firm body at 6 minutes. This foam was hard with some flex.

35 Example 43

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

70.0g Castor oil  
15.0g A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO<sub>2</sub> (20-21 wt. %) (EG-ST)  
40 10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

- 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)  
 0.4g Dipropylene glycol (33LV Catalyst)  
 0.02g Dibutyltin dilaurate (Di-10)  
 0.4g 1,8-diazabicyclo(5,4,0)-7undecane 2-ethyl hexoate, CAS # 33918-18-2  
 5 (Polycat SA102)  
 0.3g Bis(dimethyl amino propyl)methyl amine (POLYCAT 77)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight at 45 seconds to initiate, 100 seconds to gel  
 10 and cure, and 130 seconds to foam and rise. The foam had a good firm body at 6 minutes. This foam was hard with some flex.

#### Example 44

The chemicals were added to a mixing vessel in the order listed at room  
 15 temperature and thereafter thoroughly mixed:

- Side B components:  
 70.0g Castor oil  
 15.0g Polyethylene Glycol 400 (PEG400)  
 10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)  
 20 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)  
 0.4g Dipropylene glycol (33LV Catalyst)  
 0.02g Dibutyltin dilaurate (Di-10)  
 0.4g 1,8-diazabicyclo(5,4,0)-7undecane 2-ethyl hexoate, CAS # 33918-18-2  
 (Polycat SA102)  
 25 0.3g Bis(dimethyl amino propyl)methyl amine (POLYCAT 77)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight at 45 seconds to initiate, 100 seconds to gel  
 and cure, and 130 seconds to foam and rise. The foam had a good firm body at 6 minutes.  
 30 This foam was hard with some flex.

#### Example 45

The chemicals were added to a mixing vessel in the order listed at room  
 temperature and thereafter thoroughly mixed:

- 35 Side B components:  
 70.0g Castor oil  
 10.0g Polyethylene Glycol 600 (PEG600)  
 10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)  
 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)  
 40 0.36g Dipropylene glycol (33LV Catalyst)  
 0.018g Dibutyltin dilaurate (Di-10)

0.36g 1,8-diazabicyclo(5,4,0)-7undecane 2-ethyl hexoate, CAS # 33918-18-2  
(Polycat SA102)

0.27g Bis(dimethyl amino propyl)methyl amine (POLYCAT 77)

- 5                   The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight at 45 seconds to initiate, 100 seconds to gel and cure, and 130 seconds to foam and rise. The foam had a good firm body at 6 minutes. This foam was hard with some flex.

10   Example 46

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 65.0g Castor oil  
15   10.0g Linseed Oil  
15.0g Polyethylene Glycol 600 (PEG600)  
10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)  
1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)  
0.36g Dipropylene glycol (33LV Catalyst)  
20   0.018g Dibutyltin dilaurate (Di-10)  
0.36g 1,8-diazabicyclo(5,4,0)-7undecane 2-ethyl hexoate, CAS # 33918-18-2  
(Polycat SA102)  
0.27g Bis(dimethyl amino propyl)methyl amine (POLYCAT 77)

- 25                   The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight at 45 seconds to initiate, 100 seconds to gel and cure, and 130 seconds to foam and rise. The foam had a good firm body at 6 minutes. This foam was hard with some flex.

30   Example 47

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 65.0g Castor oil  
35   10.0g Linseed Oil  
15.0g Ethylene Glycol (EG)  
10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)  
1.0g Polyalkyleneoxide/methylsiloxane copolymer (Niax L6900)  
0.05g Dipropylene glycol (33LV Catalyst)  
40   0.05g Dibutyltin dilaurate (Di-10)  
0.075g Tripropylamine (TPA)

2.0g Sol C (see below)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight at 45 seconds to initiate, 100 seconds to gel and cure, and 130 seconds to foam and rise. The foam had a good firm body at 6 minutes. This foam was hard with some flex.

Preparation of Sol C:

Thoroughly mix the following components at room temperature to form a solution:

0.5g Carboxymethyl cellulose (CMC)

100.0g water

Example 48

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

65.0g Castor oil

10.0g Linseed Oil

15.0g Ethylene Glycol (EG)

10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

1.0g Niax L6900

0.175g Dipropylene glycol (33LV Catalyst)

0.075g Dibutyltin dilaurate (Di-10)

0.025g Tripropylamine (TPA)

2.0g Colloidal Silica (SNOWTEX)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight at 45 seconds to initiate, 100 seconds to gel and cure, and 130 seconds to foam and rise. The foam had a good firm body at 6 minutes. This foam was hard with some flex.

Example 49

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

70.0g Castor oil

0.025g Dibutyltin dilaurate (Di-10)

1.0g Dipropylene glycol (33LV Catalyst)

0.6g Calcium oxide (CaO)

10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

15.0g Ethylene Glycol (EG)

1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam.

#### 5 Example 50

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 65.0g Castor oil
- 10 10.0g Linseed Oil
- 15.0g A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO<sub>2</sub> (20-21 wt. %) (EG-ST)
- 10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
- 1.5g Colloidal Silica (SNOWTEX)
- 15 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 0.36g Dipropylene glycol (33LV Catalyst)
- 0.018g Dibutyltin dilaurate (Di-10)
- 0.36g 1,8-diazabicyclo(5,4,0)-7undecane 2-ethyl hexoate, CAS # 33918-18-2 (Polycat SA102)
- 20 0.27g Bis(dimethyl amino propyl)methyl amine (POLYCAT 77)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. The foam had a firm body at 6 minutes. This foam was hard with some flex.

#### 25 HARD FOAM (Examples 51-67)

##### Example 51

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- 30 Side B components:
- 40.0g P400 Resin (50:50 ratio)\*
- 5.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
- 5.0g Aluminum trihydrate (ATH)
- 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 35 0.3g Tripropylamine (TPA)
- 0.3g Dibutyltin dilaurate (Di-10)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam.

#### 40 The preparation of P400 Resin (50:50):

P400 Resin (50:50) comprises HFP prepolymer resin in PEG400 at a ratio 50:50 by weight which has been thoroughly mixed.

#### Example 52

5 The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 35.0g P400 Resin
- 5.0g 2-hydroxyethylmethacrylate (HEMA)
- 10 5.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
- 5.0g Aluminum trihydrate (ATH)
- 1.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 0.3g Tripropylamine (TPA)
- 0.3g Dibutyltin dilaurate (Di-10)

15 The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam.

#### 20 Example 53

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 10.0g HFP Resin (50:50)
- 25 20.0g Castor Oil
- 1.0g Calcium oxide (CaO)
- 5.0g Ethylene Glycol (EG)
- 5.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
- 5.0g Aluminum trihydrate (ATH)
- 30 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 0.5g Tripropylamine (TPA)
- 0.3g Dibutyltin dilaurate (Di-10)

35 The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam.

#### Example 54

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- 40 Side B components:
- 10.0g P400 Resin
- 20.0g Castor Oil



- 1.0g Calcium oxide (CaO)
- 5.0g Ethylene Glycol (EG)
- 5.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
- 5.0g Aluminum trihydrate (ATH)
- 5 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 0.5g Tripropylamine (TPA)
- 0.3g Dibutyltin dilaurate (Di-10)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate  
 10 (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam.

#### Example 55

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- 15 Side B components:
- 30.0g P400 Resin
- 30.0g Castor Oil
- 10.0g Calcium oxide (CaO)
- 1.0g 4,4-methylenebis(N,N-dimethylaniline) 10 % in maleate resin 90% by volume
- 20 (AK10)
- 1.0g 40 % by volume dibenzyl peroxide in Water (BPO Liquid)
- 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate  
 25 (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam.

#### Example 56

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- 30 Side B components:
- 10.0g HFP Resin (50:50)
- 10.0g P400 Resin
- 10.0g Castor Oil
- 1.0g Calcium oxide (CaO)
- 35 5.0g Ethylene Glycol (EG)
- 5.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
- 5.0g Aluminum trihydrate (ATH)
- 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 0.5g Tripropylamine (TPA)
- 40 0.3g Dibutyltin dilaurate (Di-10)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam.

#### Example 57

5           The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

##### Side B components:

- 10.0g Vinyl Ester Resin
- 10.0g HFP Resin (50:50)
- 10   10.0g P400 Resin
- 10.0g Castor Oil
- 1.0g Calcium oxide (CaO)
- 5.0g Ethylene Glycol (EG)
- 5.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
- 15   5.0g Aluminum trihydrate (ATH)
- 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 0.5g Tripropylamine (TPA)
- 0.3g Dibutyltin dilaurate (Di-10)

20           The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam.

#### Example 58

25           The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

##### Side B components:

- 10.0g Vinyl ester resin
- 10.0g HFP Resin (50:50)
- 10.0g Castor Oil
- 30   0.25g N'N-dimethylaniline (N'N DMA)
- 0.25g Cobalt naphthanate (NAPH)
- 1.0g Water
- 0.5g Dibutyltin dilaurate (Di-10)
- 0.5g Triethylamine (TEA)
- 35   0.6g 40 % by volume dibenzyl peroxide in Water (BPO Liquid)
- 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam.

40

Example 59

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

## Side B components:

- 5 10.0g Vinyl ester resin
- 10.0g HFP Resin(50:50)
- 10.0g Castor Oil
- 6.0g CaCO<sub>3</sub>
- 0.25g N'N-dimethylaniline (N'N DMA)
- 10 0.25g Cobalt naphthanate (NAPH)
- 3.0g Water
- 0.5g Dibutyltin dilaurate (Di-10)
- 0.5g Triethylamine (TEA)
- 0.6g 40 % by volume dibenzyl peroxide in Water (BPO Liquid)
- 15 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam was very rigid.

20

Example 60

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

## Side B components:

- 25 10.0g VE Resin
- 10.0g HFP Resin (50:50)
- 10.0g Castor Oil
- 10.0g Sol 4 (see Example 2)
- 5.0g CaCO<sub>3</sub>
- 30 0.25g N'N-dimethylaniline (N'N DMA)
- 0.25g Cobalt naphthanate (NAPH)
- 0.5g Dibutyltin dilaurate (Di-10)
- 0.5g Triethylamine (TEA)
- 0.6g 40 % by volume dibenzyl peroxide in Water (BPO Liquid)
- 35 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam was very rigid.

40

Example 61

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 5 10.0g Vinyl ester resin
- 10.0g HFP Resin(50:50)
- 10.0g Castor Oil
- 10.0g Sol 4 (see Example 2)
- 10.0g Ethylene Glycol (EG)
- 10 10.0g CaCO<sub>3</sub>
- 0.25g N,N-dimethylaniline (N,N DMA)
- 0.25g Cobalt naphthanate (NAPH)
- 0.5g Dibutyltin dilaurate (Di-10)
- 0.5g Triethylamine (TEA)
- 15 0.6g 40 % by volume dibenzyl peroxide in Water (BPO Liquid)
- 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam  
20 was very rigid.

Example 62

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- 25 Side B components:
- 10.0g Vinyl ester resin
- 10.0g HFP Resin(25:75)\*\*
- 10.0g Castor Oil
- 0.5g Sol 4 (see Example 2)
- 30 0.5g Dibutyltin dilaurate (Di-10)
- 0.5g Triethylamine (TEA)
- 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam  
35 was very rigid and had large cells.

The preparation of HFP Resin (25:75):

The following components were thoroughly mixed to form a mixture.

- 40 25% by weight HFP polyester resin
- 75% by weight 2-hydroxyethylmethacrylate (HEMA)

To every 30.0g of above mixture, 3.0g of 40% by volume dibenzyl peroxide (BPO Liquid) was added and thoroughly mixed to form the resin.

Example 63

5           The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 10.0g Vinyl ester resin
- 10.0g HFP Resin (25:75)
- 10   10.0g Castor Oil
- 11.0g Polyethylene Glycol 400 (PEG400)
- 0.5g Dibutyltin dilaurate (Di-10)
- 0.5g Triethylamine (TEA)
- 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

15           The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam was very rigid with large cells.

20   Example 64

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 10.0g Vinyl Ester Resin
- 25   10.0g HFP Resin (25:75)
- 10.0g Castor Oil
- 7.0g Polyethylene Glycol 400 (PEG400)
- 0.5g Dibutyltin dilaurate (Di-10)
- 0.5g Triethylamine (TEA)
- 30   0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam was very rigid with large cells.

35   Example 65

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 40   15.0g HFP Resin (25:75)
- 15.0g Castor Oil

- 15.0g Polyethylene Glycol 400 (PEG400)
- 1.0g Sol 4 (see Example 2)
- 5.0g Ethylene Glycol (EG)
- 0.075g Dibutyltin dilaurate (Di-10)
- 5 0.075g Triethylamine (TEA)
- 0.5g Polyalkyleneoxide/methylsiloxane copolymer (Niax L5340)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam  
10 was very rigid with large cells.

#### Example 66

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- 15 Side B components:
- 7.5g HFP Resin (25:75)
- 7.5g PEG400 Resin (50:50)
- 15.0g Castor Oil
- 15.0g Polyethylene Glycol 400 (PEG400)
- 20 1.0g Sol 4 (see Example 2)
- 5.0g Ethylene Glycol (EG)
- 0.25g Dibutyltin dilaurate (Di-10)
- 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

25 The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam was very rigid with large cells.

#### Example 67

30 The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- Side B components:
- 15.0g HFP Resin (25:75)
- 15.0g PEG400 Resin (50:50)
- 35 30.0g Castor Oil
- 30.0g Polyethylene Glycol 400 (PEG400)
- 2.0g Sol 4 (see Example 2)
- 10.0g Ethylene Glycol (EG)
- 0.5g Dibutyltin dilaurate (Di-10)
- 40 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam was very rigid with large cells.

## 5 ADHESIVE FOAM (Examples 68-77)

### Example 68

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- 10 Side B components:
  - 10.0g P2 Resin
  - 10.0g PEG400 Resin (50:50)
  - 10.0g Castor Oil
  - 0.3g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 15 0.5g 40 % by volume dibenzyl peroxide in Water (BPO Liquid)
- 0.25g Dibutyltin dilaurate (Di-10)

- The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam
- 20 had good cell structure and remained tacky for 10-15 min.

### Example 69

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

- 25 Side B components:
  - 10.0g HFP Resin (50:50)
  - 10.0g PEG400 Resin (50:50)
  - 10.0g Castor Oil
  - 0.7g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 30 0.7g 40 % by volume dibenzyl peroxide in Water (BPO Liquid)
- 0.25g Dibutyltin dilaurate (Di-10)

- The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam
- 35 had good cell structure and remained tacky for 10-15 min.

### Example 70

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 10.0g Vinyl Ester Resin
- 10.0g P2 Resin\*
- 10.0g PEG400 Resin (50:50)
- 10.0g Castor Oil
- 5 0.5 Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 1.0 40 % by volume dibenzyl peroxide in Water (BPO Liquid)
- 0.25g Dibutyltin dilaurate (Di-10)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam had good cell structure and remained tacky for 10-15 min.

#### Example 71

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

Mix D is initially prepared by adding the following components in the order listed and mixing thoroughly:

- 70.0g Castor Oil
- 0.025g Dibutyltin dilaurate (Di-10)
- 0.025g Tripropylamine (TPA)
- 0.7g Dipropylene glycol (33LV Catalyst)
- 1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)
- 10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)
- 20.0g Ethylene Glycol (EG)
- 2.0g Fumed Silica

Thereafter the side B solution is formed by mixing the following components in a mixing vessel.

- 22.5g Mix D
- 1.0g PEG400 Resin (50:50)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam had good cell structure and remained tacky for 10-15 min.

#### Example 72

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:



Mix E was prepared by adding the following components into a mixing vessel in the order listed and thoroughly mixed.

70.0g Castor Oil

0.050g Dibutyltin dilaurate (Di-10)

5 0.7g Dipropylene glycol (33LV Catalyst)

1.0g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

20.0g Ethylene Glycol (EG)

2.0g Fumed Silica

10

Thereafter the side B solution was prepared by thoroughly mixing 22.5g of Mix E with 1g of PEG400 Resin (50:50).

15 The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam had good cell structure and remained tacky for 10-15 min.

#### Example 73

20 The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

70.0g Castor Oil

0.025g Dibutyltin dilaurate (Di-10)

0.075g Dipropylene glycol (33LV Catalyst)

25 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

15.0g Ethylene Glycol (EG)

30 The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam had good cell structure and remained tacky for 10-15 min.

#### Example 74

35 The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

90.0g Castor Oil

0.8g Dipropylene glycol (33LV Catalyst)

0.075g Dibutyltin dilaurate (Di-10)

40 0.5g Polyalkyleneoxide/methylsiloxane copolymer (Niax L6900)

10.0g FYROL™ (tri(2-chloroethyl)phosphate flame retardant)

1.5g Sol C

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam had good cell structure and remained tacky for 10-15 min.

5

Example 75

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 10 65.0g Castor Oil
- 25.0g Linseed Oil
- 0.075g Dibutyltin dilaurate (Di-10)
- 0.8g Dipropylene glycol (33LV Catalyst)
- 0.5g Polyalkyleneoxide/methylsiloxane copolymer (Niax L6900)
- 15 10.0g FYROL™ (tri(2-chloroethyl)phosphate flame retardant)
- 1.5g Sol C

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam had good cell structure and remained tacky for 10-15 min.

20

Example 76

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

Side B components:

- 25 65.0g Castor Oil
- 25.0g Linseed Oil
- 0.075g Dibutyltin dilaurate (Di-10)
- 0.8g Dipropylene glycol (33LV Catalyst)
- 30 0.5g Polyalkyleneoxide/methylsiloxane copolymer (Niax L6900)
- 10.0g FYROL™ (tri(2-chloroethyl)phosphate flame retardant)
- 1.5g Colloidal Silica (SNOWTEX)

The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam had good cell structure and remained tacky for 10-15 min.

35

Example 77

The chemicals were added to a mixing vessel in the order listed at room temperature and thereafter thoroughly mixed:

40

## Side B components:

70.0g Castor Oil

0.025g Dibutyltin dilaurate (Di-10)

0.075g Dipropylene glycol (33LV Catalyst)

5 0.5g Polyalkylene/oxidemethylsiloxane copolymer (Niax Silicone L5340)

10.0g FYROL™ (tri (2-chloroethyl)phosphate flame retardant)

15.0g A mixture of ethylene glycol (80-79 wt. %) and amorphous SiO<sub>2</sub> (20-21 wt. %) (EG-ST)

10 The side B solution was mixed with 4,4'-diphenyl methane diisocyanate (LUPRANATE™) (MDI) at 1:1 ratio by weight to form a polyurethane foam. This foam had good cell structure and remained tacky for 10-15 min.

Preparation of P-2 Resin, P-4 Resin, and P-6 Resin:

15 P-2 Resin, P-4 Resin, and P-6 Resin was prepared by thoroughly mixing the following components as indicated:

Co Polymer PH:

33.33g Polyethylene Glycol 200 (PEG200)

66.66g 2-hydroxyethylmethacrylate (HEMA)

20

P-2 Resin is a mix of 25 wt. % HFP polyester resin and 75 wt. % co-polymer PH.

P-4 Resin is the same as P-2 Resin except that PEG400 is substituted for PEG200.

P-6 Resin is the same as P-2 Resin except that PEG600 is substituted for PEG200.

25 Preparation of HFP polyester resin:

A high flash point polyester resin was prepared by mixing the following components in a four-neck reaction flask with stirring under a nitrogen atmosphere:

880.6 g propylene glycol

366.30 g fumaric acid

30 206.32 g maleic anhydride

779.11 g phthalic anhydride

The above components were heated at 150° C. for about 3 to 5 hours. The temperature was raised to 180° C. and maintained for about 3 hours, removing water produced during the reaction through a trap. The reaction mixture was placed under vacuum  
35 (25 in. Hg) and the temperature was raised to 190° C. for about 4 to 5 hours. The resulting solid was designated Solid Resin #1.

In the above examples, the ratios and the components of the solutions can be varied. For example, cane syrup, sugar syrup, or combinations thereof can be employed as a

carbohydrate component. Similarly, ethylene glycol can be substituted with or mixed with PEG200, PEG400, PEG600, or combinations thereof. Further, cross linking agents such as Pentaerythritol tetraacrylate (PENTA), PEG dimethacrylate (200,400,600), or any cross linking agent that suitable for water blowing foam may be employed. Catalyst, such as  
 5 LV33, TEA, TPA, or any catalyst suitable for water blowing systems, or any combination thereof, may also be employed. Orange oil was also added as a scent. Further, any silicone surfactant suitable with water may be employed.

#### Chemicals Employed In The Above Examples:

- 10 AntaroX 25-R-2  
Company --Rhodia --CN7500, Cranbury, NY 08512  
Main components -- Propoxylated polyoxyethylene 99.5%
- Niax Silicone L-5340  
15 Company --Witco Corporation, OSI Specialites group, Sistersville, WV 26175  
Main components- Polyalkyleneoxide/methylsiloxane copolymer
- Niax Silicone L-6900  
Company --Witco Corporation, OSI Specialites group, Sistersville, WV 26175  
20 Polyalkyleneoxide/methylsiloxane copolymer
- Aminified Sugar (0.1 % of microwave initiator added to diethyl cyclohexylamine)  
Chemical name Diethyl cyclohexylamine CAS# 91-65-6  
Main Supplier -- Air Products- 7201 Hamilton Blvd. Allentown PA18195  
25 Pure Cane Sugar  
Company --Savannah Foods -- Savannah GA USA  
PCS Molasses (trade name)
- 30 Corn Syrup  
Company --Sunlight Foods Inc, Sanford, FL 32771  
Tradename HFCS42
- FYROL™ CEF (FYROL™)  
35 Company-- Akzo Nobel Chemicals, 5 Livingston Ave., Dobbs Ferry, NY 10522  
Main Components—Ethanol, 2-chloro-phosphate CAS#115-95-8
- Cadox 40E (BPO Liquid)  
Company -- Akzo Nobel Chemicals—300 Riverside parkway, Chicago, IL, 60606  
40 Components- 40 % by volume dibenzyl peroxide in Water
- Snowtex

- Colloidal Silica  
Company --Nissan Chemical industries Japan
- ZF-20  
5 2,2'-oxybis(N,N-dimethylethanamine) (CAS No. 3033-62-3)  
Company – Huntsman Company
- Dibutyltin Laurate  
CAS # 77-58-7  
10
- POLYCAT 77 Catalyst  
Bis(dimethyl amino propyl)methyl amine, CAS # 3855-32-1  
Company –Air Products –7201 Hamilton Blvd., Allentown PA 18195
- 15 Polycat SA102 Catalyst (POLYCAT 102)  
1,8-diazabicyclo(5,4,0)-7undecane 2-ethyl hexoate, CAS # 33918-18-2  
Company –Air Products –7201 Hamilton Blvd., Allentown PA 18195
- 20 Dabco 33LV Catalyst  
Company –Air Products –7201 Hamilton Blvd., Allentown PA 18195  
Components—Dipropylene glycol CAS#2526-71-8  
Triethelenediamine CAS#280-57-9
- 25 Fumed Silica  
Wacker HDK (trade name)  
Company –Wacker made in Munich Germany
- 30 Granulated Sugar (crushed), Sugar Crystals  
(DIXIE CRYSTALS PURE SUGAR)  
Company - Dixie Crystals Food Service Savannah GA 31405
- EG-ST  
Ethylene glycol (80-79 wt. %) and amorphous silica (20-21 wt. %)  
35 Nissan Chemical Industries, Ltd., Sodegaura Plant, Japan
- Hawks FG91 (AK10)  
Company - Hawk Chemical Company  
4,4-methylenebis(N,N-dimethylaniline) 10 % in maleate resin 90% by volume, CAS #101-  
40 61-1

It should be understood, of course, that the foregoing relates only to preferred embodiments of the present invention and that numerous modifications or alterations may be made therein without departing from the spirit and the scope of the invention.

## WHAT IS CLAIMED IS:

1. A polyurethane foam comprising the reaction product of a polyol selected from a fatty acid, a glycol, a mineral oil, a carbohydrate, or a combination thereof with a polyisocyanate in the presence of a catalyst and at least one blowing agent.

5 2. The foam of claim 1, wherein the fatty acid is selected from palmitic, stearic, oleic linoleic, myristic, arachidic, ricinoleic, or a combination thereof.

3. The foam of claim 1, wherein the glycol is selected from glycol, ethylene glycol, a polyethylene glycol, diethylene glycol, dipropylene glycol, propylene glycol, hexylene glycol, neopentyl glycol, bisphenol A, 2-methyl propanediol, 10 trimethylolpropane, 1,4-butane diol, or a combination thereof.

4. The foam of claim 3, wherein the polyethylene glycol has a molecular weight range between about 200 and about 600.

5. The foam of claim 1, wherein the carbohydrate is a polyhydric alcohol having the general formula:

15 
$$\text{CH}_2\text{OH}(\text{CHOH})_n\text{CH}_2\text{OH},$$
  
wherein n is selected from 2 to 5.

6. The foam of claim 1, wherein the polyhydric alcohol is selected from sucrose, glucose, fructose kojibiose, turanose, isomaltose, maltose, dextrose, glyceride, and combinations thereof.

20 7. The foam of claim 1, wherein the polyisocyanate is selected from hexamethylene diisocyanate, toluene 2-4 diisocyanate, naphthalene 1,5 diisocyanate, diphenylmethane 2,4' diisocyanate, 4,4' diphenyl methane diisocyanate, or a combination thereof.

25 8. The foam of claim 1, wherein the blowing agent is water, a low-boiling inert liquid, carbon dioxide, air, or a combination thereof.

9. The foam of claim 8, wherein the low-boiling inert liquid is selected from a chlorofluorocarbon, a hydrochlorofluorocarbon, a hydrofluorocarbon, a pentane, chloroform, or a combination thereof.

30 10. A polyurethane foam comprising the reaction product of a polyol selected from a vegetable oil, a mineral oil, a glycol, a syrup, or a combination thereof with a polyisocyanate in the presence of a catalyst and at least one blowing agent.

11. The foam of claim 10, wherein the vegetable oil is selected from canola oil, sesame oil, sunflower oil, soybean oil, palm oil, castor oil, coconut oil, cotton seed oil, olive oil, apricot oil, avocado oil, safflower oil, linseed oil, coconut oil, corn oil, babassu oil, tung oil, perilla oil, oiticica oil, a hydrogenated derivative thereof, or a combination thereof.

5 12. The foam of claim 10, wherein the glycol is selected from glycol, ethylene glycol, a polyethylene glycol, diethylene glycol, dipropylene glycol, propylene glycol, hexylene glycol, neopentyl glycol, bisphenol A, 2-methyl propanediol, trimethylolpropane, 1,4-butane diol, or a combination thereof.

10 13. The foam of claim 12, wherein the polyethylene glycol has a molecular weight range between about 200 and about 600.

14. The foam of claim 10, wherein the syrup is selected from maple syrup, honey, corn syrup, cane syrup, golden syrup, molasses, sorghum, beet syrup, or a combination thereof.

15 15. The foam of claim 10, wherein the syrup comprises sucrose, glucose, fructose kojibiose, turanose, isomaltose, maltose, dextrose, glycerol, and combinations thereof.

20 16. The foam of claim 10, wherein the polyisocyanate is selected from hexamethylene diisocyanate, toluene 2-4 diisocyanate, naphthalene 1,5 diisocyanate, diphenylmethane 2,4' diisocyanate, 4,4' diphenyl methane diisocyanate, or a combination thereof.

17. The foam of claim 10, wherein the blowing agent is water, a low-boiling inert liquid, carbon dioxide, air, or a combination thereof.

25 18. The foam of claim 17, wherein the low-boiling inert liquid is selected from a chlorofluorocarbon, a hydrochlorofluorocarbon, a hydrofluorocarbon, a pentane, chloroform, or a combination thereof.

19. A method for producing polyurethane foam compositions comprising:  
producing a polyol blend comprising a fatty acid, a glycol, a mineral oil, a carbohydrate, or a combination thereof; and

30 reacting the polyol blend with a polyisocyanate blend in the presence of a catalyst and a blowing agent.

20. The method as claimed in claim 19, wherein the polyol blend and the polyisocyanate blend are reacted with one another in equal parts by weight.

21. The method of claim 19, wherein the fatty acid is selected from palmitic, stearic, oleic linoleic, myristic, arachidic, ricinoleic, or a combination thereof.

5 22. The method of claim 19, wherein the glycol is selected from glycol, ethylene glycol, a polyethylene glycol, diethylene glycol, dipropylene glycol, propylene glycol, hexylene glycol, neopentyl glycol, bisphenol A, 2-methyl propanediol, trimethylolpropane, or a combination thereof.

10 23. The method of claim 22, wherein the polyethylene glycol has a molecular weight range between about 200 and about 600.

24. The method of claim 19, wherein the carbohydrate is a polyhydric alcohol having the general formula:



wherein n is selected from 2 to 5.

15 25. The method of claim 19, wherein the polyhydric alcohol is selected from sucrose, glucose, fructose kojibiose, turanose, isomaltose, maltose, dextrose, glyceride, and combinations thereof.

26. The method of claim 19, wherein the polyisocyanate is selected from hexamethylene diisocyanate, toluene 2-4 diisocyanate, naphthalene 1,5 diisocyanate, 20 diphenylmethane 2,4' diisocyanate, 4,4' diphenyl methane diisocyanate, or a combination thereof.

27. The method of claim 19, wherein the blowing agent is water, a low-boiling inert liquid, carbon dioxide, air, or a combination thereof.

28. The method of claim 27, wherein the low-boiling inert liquid is selected 25 from a chlorofluorocarbon, a hydrochlorofluorocarbon, a hydrofluorocarbon, a pentane, chloroform, or a combination thereof.

29. A method for producing polyurethane foam compositions comprising:

producing a polyol blend comprising a vegetable oil, a mineral oil, a glycol, a syrup, or a combination thereof ; and

30 reacting the polyol blend with a polyisocyanate blend in the presence of a catalyst and a blowing agent.



30. The method of claim 29, wherein the vegetable oil is selected from canola oil, sesame oil, sunflower oil, soybean oil, palm oil, castor oil, coconut oil, cotton seed oil, olive oil, apricot oil, avocado oil, safflower oil, linseed oil, coconut oil, corn oil, babassu oil, tung oil, perilla oil, oiticica oil, a hydrogenated derivative thereof, or a combination thereof.

31. The foam of claim 29, wherein the glycol is selected from glycol, ethylene glycol, a polyethylene glycol, diethylene glycol, dipropylene glycol, propylene glycol, hexylene glycol, neopentyl glycol, bisphenol A, 2-methyl propanediol, trimethylolpropane, or a combination thereof.

32. The foam of claim 31, wherein the polyethylene glycol has a molecular weight range between about 200 and about 600.

33. The foam of claim 29, wherein the syrup is selected from maple syrup, honey, corn syrup, cane syrup, golden syrup, molasses, sorghum, beet syrup, or a combination thereof.

34. The foam of claim 29, wherein the syrup comprises sucrose, glucose, fructose kojibiose, turanose, isomaltose, maltose, dextrose, glycerol, and combinations thereof.

35. The foam of claim 29, wherein the polyisocyanate is selected from hexamethylene diisocyanate, toluene 2-4 diisocyanate, naphthalene 1,5 diisocyanate, diphenylmethane 2,4' diisocyanate, 4,4' diphenyl methane diisocyanate, or a combination thereof.

36. The foam of claim 29, wherein the blowing agent is water, a low-boiling inert liquid, carbon dioxide, air, or a combination thereof.

37. The foam of claim 36, wherein the low-boiling inert liquid is selected from a chlorofluorocarbon, a hydrochlorofluorocarbon, a hydrofluorocarbon, a pentane, chloroform, or a combination thereof.

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
27 September 2001 (27.09.2001)

PCT

(10) International Publication Number  
**WO 01/70842 A3**

(51) International Patent Classification<sup>7</sup>: C08G 18/36.  
18/32, C08J 9/00

(21) International Application Number: PCT/US01/08888

(22) International Filing Date: 20 March 2001 (20.03.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:  
60/190,642 20 March 2000 (20.03.2000) US

(71) Applicant: KT HOLDINGS, LLC [US/US]; 2137E Flin-  
stone Drive, Tucker, GA 30084 (US).

(72) Inventors: KATOOT, Mohammad, W. (deceased). KA-  
TOOT, Ahmed, M.: 1011 Lulworth Lane, Lawrenceville,  
GA 30044 (US).

(74) Agents: ARNOLD, Jeffrey, B. et al.: Kilpatrick Stockton  
LLP, 2400 Monarch Tower, 3424 Peachtree Road, Atlanta,  
GA 30326 (US).

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU,  
AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ,  
DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR,  
HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,  
LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ,  
NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM,  
TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.

(84) Designated States (*regional*): ARIPO patent (GH, GM,  
KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian  
patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European  
patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE,  
IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF,  
CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

**Published:**

— with international search report

(88) Date of publication of the international search report:  
31 January 2002

*For two-letter codes and other abbreviations, refer to the "Guid-  
ance Notes on Codes and Abbreviations" appearing at the begin-  
ning of each regular issue of the PCT Gazette.*

(54) Title: POLYURETHANE FOAMS

(57) Abstract: Polyurethane foams are formed as the reaction product of a polyol selected from a fatty acid, a glycol, a mineral oil, a carbohydrate, or a combination thereof with a polyisocyanate in the presence of a catalyst and at least one blowing agent. Also, polyurethane foams are formed as the reaction product of a polyol selected from a vegetable oil, a mineral oil, a glycol, a syrup, or a combination thereof with a polyisocyanate in the presence of a catalyst and at least one blowing agent. A method for producing polyurethane foam compositions is directed to producing a polyol blend comprising a fatty acid, a glycol, a mineral oil, a carbohydrate, or a combination thereof, and reacting the polyol blend with a polyisocyanate blend in the presence of a catalyst and a blowing agent. Also, a method for producing polyurethane foam compositions is directed to producing a polyol blend comprising a vegetable oil, a mineral oil, a glycol, a syrup, or a combination thereof, and reacting the polyol blend with a polyisocyanate blend in the presence of a catalyst and a blowing agent.

WO 01/70842 A3

# INTERNATIONAL SEARCH REPORT

In **ational Application No**  
PCT/US 01/08888

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 7 C08G18/36 C08G18/32 C08J9/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 7 C08G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, PAJ, EPO-Internal

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 451 615 A (BIRCH ADRIAN J) 19 September 1995 (1995-09-19)  column 2, line 55 -column 4, line 17; claims 1,9,11,13,15; example 2; table 2 ---	1-4, 7-13, 16-23, 26-32, 35-37
X	US 2 833 730 A (BARTHEL JR ERIC) 6 May 1958 (1958-05-06)  column 2, line 14 - line 48; claims 1,4; examples 1-4,7 --- -/--	1,3,4,7, 8,10-13, 16,17, 19,22, 23,26, 27, 29-32, 35,36

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*&\* document member of the same patent family

Date of the actual completion of the international search

26 October 2001

Date of mailing of the international search report

05/11/2001

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Angiolini, D

# INTERNATIONAL SEARCH REPORT

In ternational Application No

PCT/US 01/08888

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB 1 032 365 A (BIBBY & SONS LTD J) 8 June 1966 (1966-06-08)  page 2, line 7 -page 3, line 22; claims 13,14; example 9; table ---	1-4,7,8, 10-13, 16,17, 19-23, 26,27, 29-32, 35,36
X	WO 97 07150 A (STIELAU MARTIN ERNST) 27 February 1997 (1997-02-27)  page 1; claims 1,7; examples 1,3 ---	1,3,7, 10,12, 16,19, 22,26, 29,31,35
A	US 4 201 847 A (KLEIMANN HELMUT ET AL) 6 May 1980 (1980-05-06)	1,2,5, 7-9, 19-21, 24,26-28
X	column 6, line 17 - line 52; claims 1,2; examples 8,9  -----	10,11, 16-18, 29,30, 35-37

# INTERNATIONAL SEARCH REPORT

Information on patent family members

Int. Application No

PCT/US 01/08888

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 5451615	A	19-09-1995	AU 3833795 A BR 9509500 A CA 2201586 A1 CN 1161705 A ,B CZ 9701192 A3 EP 0787165 A2 HU 77801 A2 JP 11500467 T PL 319832 A1 TR 965332 A2 WO 9612759 A2	15-05-1996 14-10-1997 02-05-1996 08-10-1997 13-08-1997 06-08-1997 28-08-1998 12-01-1999 01-09-1997 21-06-1996 02-05-1996
US 2833730	A	06-05-1958	NONE	
GB 1032365	A	08-06-1966	DE 1495170 A1	20-02-1969
WO 9707150	A	27-02-1997	PT 101761 A PT 101863 A AT 198899 T AU 704532 B2 AU 6758396 A BR 9610395 A DE 69611679 D1 EP 0846136 A1 JP 11512122 T WO 9707150 A1 US 6051623 A	31-03-1997 31-12-1997 15-02-2001 29-04-1999 12-03-1997 09-05-2000 01-03-2001 10-06-1998 19-10-1999 27-02-1997 18-04-2000
US 4201847	A	06-05-1980	DE 2307589 A1 AT 331037 B AT 128074 A AU 6555674 A BE 811005 A1 CA 1037217 A1 CH 592695 A5 DK 141851 B ES 423291 A1 FR 2218363 A1 GB 1420293 A IE 38896 B1 IT 1002917 B JP 1187831 C JP 49114695 A JP 58018368 B LU 69391 A1 NL 7401994 A ,B, SE 416655 B US 4254228 A	05-09-1974 26-07-1976 15-10-1975 14-08-1975 14-08-1974 29-08-1978 15-11-1977 30-06-1980 01-06-1976 13-09-1974 07-01-1976 21-06-1978 20-05-1976 30-01-1984 01-11-1974 12-04-1983 01-10-1974 20-08-1974 26-01-1981 03-03-1981